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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1. **Progress in analytical chemistry.** I. P. Alimarin (V.I. Vernadsky Inst., Akad. Nauk SSSR, Moscow). *Chem. Listy*, 1960, **54** (5), 462–466.—A review.

2. **Equipment of the organic chemistry laboratory for gas chromatography.** W. Simon (Eidgenössischen Tech. Hochsch., Zürich, Switzerland). *Chimia*, 1960, **14** (6), 189–201.—This review includes an introduction to the theoretical and instrumental foundations of gas chromatography, an account of its development and qualitative, quantitative and preparative applications, and a discussion of commercial gas-chromatographic equipment. The products of 37 companies are listed. (98 references.)
E. G. CUMMINS

3. **Extraction in analytical chemistry.** B. Trémillon (École Supér. de Phys. et de Chim., rue Vauquelin, Paris). *Bull. Soc. Chim. France*, 1960, (6), 1011–1013.—A discussion is presented, with examples, of the analytical uses of extraction of inorganic compounds and organometallic complexes by H_2O-immiscible solvents, with special reference to (*i*) the behaviour of the organic phase and (*ii*) selective extraction or separation by controlled displacement of equilibria in the aq. phase.
W. J. BAKER

4. **Toxicological analysis.** A. S. Curry (Home Office Forensic Sci. Lab., Harrogate, England). *J. Pharm. Pharmacol.*, 1960, **12** (6), 321–339.—This is a review, with 120 references, of developments in the last five years. (*Cf, Anal. Abstr.*, 1956, **3**, 1146.)
A. R. ROGERS

5. **Radio-frequency methods in analytical chemistry.** M. F. C. Ladd and W. H. Lee (Battersea Coll. of Technol., London, England). *Talanta*, 1960, **4** (4), 274–291 (in English).—The theory of radio-frequency measurements of analytical importance is discussed, and suitable types of equipment and applications are reviewed. (166 references.)
W. T. CARTER

6. **The origin of nuclei in precipitation reactions.** R. B. Fischer (Dept. of Chem., Indiana Univ., Bloomington, U.S.A.). *Anal. Chim. Acta*, 1960, **22** (6), 501–508 (in English).—The pretreatment of water used to dissolve $BaCl_2$ is shown to affect the particle size of $BaSO_4$ subsequently pptd. For a 24-hr.-old $BaCl_2$ soln., typical particle sizes in ordinary laboratory distilled water, in the same after standing for 24 hr. in Pyrex glass, in water distilled in an all-Pyrex-glass system and in fresh conductivity water are, respectively, 16, 45, 56 and 68 μ. It is suggested that these phenomena are due to nucleation on sites already present in the water, which are fewer with purer water and which can be removed by adsorption. It is shown that the impurities in analytical-grade chemicals are more than sufficient to provide nucleation sites, other possible sources being containers and colloidal impurities.
R. M. S. HALL

7. **Effect of form of reagent on particle sizes of precipitates.** R. B. Fischer (Dept. of Chem., Indiana Univ., Bloomington, U.S.A.). *Anal. Chim. Acta*, 1960, **22** (6), 508–514 (in English).—The particle size of ppt. formed when one reagent is in solid form is, in general, one-third to one-tenth of the size obtained when both reagents are in soln. This is illustrated by pptn. of the sulphates of Pb, Ca, Ba and Sr, the fluorides of Ca and Pb, nickel dimethylglyoxime and Ag_2CrO_4. Investigation showed that the presence of a recently dissolved inert electrolyte exerts a variable effect on the particle size of the ppt. These effects are attributed to an increase in nucleation sites provided by the solid reagent, and this is further increased when the reagent is ground.
R. M. S. HALL

8. **Omega chrome green BLL: a new analytical reagent.** A. A. A. El Raheem, A. S. Moustafa and A.-A. Amin (Publ. Health Lab. and Nat. Res. Centre, Cairo, Egypt). *Z. anal. Chem.*, 1960, **175** (1), 19–29 (in English).—Copper, Pb, Th and Al can be titrated with EDTA in acid soln., with Omega chrome green BLL (C.I. Mordant Green 29) (**I**) as indicator. *Procedure for Cu*—To the sample soln. are added a few drops of indicator (0·1 g of **I** in 100 ml of twice-distilled H_2O), 15 ml of twice-distilled H_2O and 3 ml of 0·01 M HNO_3. From a burette is added an excess of 0·01 M EDTA (disodium salt) and the yellow soln. is titrated with 0·01 M $CuSO_4$ until a violet colour appears. *For Pb*—To the soln. containing $\not< 10$ mg of Pb per ml are added a few drops of **I** and 3 ml of 0·01 M HNO_3, and the soln. is titrated to a yellow end-point. *For Th*—To the soln. are added 3 ml of buffer (**II**) (pH 3·45) (25 ml of 0·2 M Na acetate and 475 ml of 0·2 M acetic acid), a few drops of **I** and 2 ml of ethanol. The mixture is warmed to 60° and titrated to a yellow end-point. *For Al*—To the acid sample soln. are added an excess of 0·1 M EDTA and $\simeq 2$ ml of **II**; the mixture is boiled for $\simeq 10$ min., then cooled in ice to 1° to 3°. After addition of 3 ml of ethanol and a few drops of **I** the soln. is titrated with 0·01 M thorium nitrate. **I** is also recommended as a colour reagent for the spectrophotometric determination of traces of Cu in the presence of a 10-fold excess of Ca, Cd, Co, Mg, Mn and Sr; and for the determination of as little as 0·02 p.p.m. of V in the presence of Al, Ca, Cd, Mg, U, Ni and Sr; the interference of Fe^{3+} is avoided by the addition of a few crystals of ascorbic acid. The results are tabulated and the standard deviation and probable errors of the titrimetric determination of these metals are 11 μg and 7 μg for Cu, 20 μg and 17 μg for Th, 14 μg and 9 μg for Pb, and 14 μg and 9 μg for Al.
B. B. BAUMINGER

9. Mixed indicators of methyl red with sulphonated phthalocyanines. T. P. Sastry and S. A. Pratt, III (Chem. Dept., Texas Southern Univ., Houston, U.S.A.). *Z. anal. Chem.*, 1960, **174** (5), 359–364 (in English).—Copper, cobalt and magnesium phthalocyanine-4,4′,4″,4‴-tetrasulphonates were investigated as components of mixed indicators. When used with methyl red all improved the end-point, giving a green to red colour change. A ratio of 2 parts of methyl red to 3 parts of copper phthalocyanine-4,4′,4″,4‴-tetrasulphonate is recommended for all acid - base titrations for which methyl red is normally used.　W. T. CARTER

10. Metallochromic indicators. IX. Indophenol-complexones. V. Svoboda, L. Dorazil and J. Körbl (Spolana Neratovice, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (4), 1037–1043.—The preparation and acid - base and metallochromic properties of a new group of indicators are described and their extinction coeff. were measured. With the use of these indicators the following complexometric titrations can be carried out in chloroacetate buffer soln.—Th^{4+} (5 to 100 mg) at pH 3·3 to 3·5; Fe^{3+} (1 to 30 mg) at 60°; Bi^{3+} (4 to 40 mg) at pH 2·8; and Sc^{3+} (1 to 40 mg) at pH 3·5. The sharpest colour changes were obtained with the use of 2,6-dibromophenolindo-o-cresol-6′-methyl-aminediacetic acid (Indoferron).　J. ZÝKA

11. Complexometric and argentimetric titrations using chemiluminescent indicators. L. Erdey and I. Buzás (Tech. Univ., Inst. for General Chemistry, Budapest). *Anal. Chim. Acta*, 1960, **22** (6), 524–531 (in English).—The mechanisms of chemilumi-nescence of lucigenin (5,5′-biacridyl dimethonitrate) and luminol (3-aminophthalhydrazide) are compared, and the use of these compounds as indicators in the complexometric determination of Cu^{II}, Pb and Hg^{II}, and for the standardisation of EDTA soln., is described. Lucigenin can be used as an adsorption indicator for the argentimetric determination of I^-, even in the presence of Br^- and Cl^-; CN^-, SCN^-, $Fe(CN)_6{}^{3-}$ and $CrO_4{}^{2-}$ interfere.
　R. M. S. HALL

12. A heterometric study of the 1,10-phenan-throline compounds with platinum, palladium and gold halides and their analytical application. M. Bobtelsky and M. M. Cohen (Dept. Inorg. and Anal. Chem., Hebrew Univ., Jerusalem). *Anal. Chim. Acta*, 1960, **22** (5), 485–495.—The apparatus and technique have been described before (*Ibid.*, 1959, **20**, 15, 227 and 357). By changing the composition of the test soln., six different final compounds were obtained for each metal at the first maximum optical density points of the curves, and each compound was suitable for the determination of the metal. Full details of the titrations are given in tabular form. Analytical errors based on these titrations were <1%. With papaverine as reagent, the reactions were approx. four times as sensitive, and in almost all cases only one final compound was formed.　R. M. S. HALL

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

13. Detection of ultra-micro quantities of inorganic cations. D. Exley (Boots Pure Drug Co., Nottingham, England). *Photoelect. Spectr. Gr.*

Bull., 1959, (12), 322–327.—Up to a 50-fold increased sensitivity in flame photometry was obtained by burning a 90% acetone solution of the sample in an oxy-hydrogen flame; the addition of 5% of HCl ensures that Na^+, K^+, Ca^{2+} and Mg^{2+} are completely in solution. The electronics of a Beckman flame photometer were modified to give greatly enhanced sensitivity, and with the combined technique it was possible to detect 3×10^{-13} g of Na^+ and 10^{-10} g of Mg^{2+}. This technique was applied to the determination of 1 to 10 mμg of Na^+, K^+, Ca^{2+} and Mg^{2+} in 0·2 to 0·5-μl samples of inner-ear fluids of cats and guinea-pigs.　P. T. BEALE

14. Differential thermal analysis of hydroxides in reducing atmosphere. W. Lodding and L. Hammell (Bureau of Mineral Res., Rutgers, State Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 657–662.—A new high temperature - pressure vacuum furnace is used with the heating element outside the tube that forms the pressure vessel. No water cooling is required. Gas flows through the sample and the reference material, and the two gas streams leave separately. This enables gas analysis of the reaction products to be carried out while differential thermal analysis is in progress. Gibbsite [$Al(OH)_3$] can be determined in the presence of hydrated iron oxides. Thermograms for various mixtures of aluminium and iron oxides are presented and discussed.　D. C. ARMSWORTH

15. Paper chromatography of inorganic ions in nitrate media. II. Separation of selenium, tellurium and polonium, and of lead-210, bismuth-210 and polonium. M. C. Levi and J. Danon (Brazilian Centre for Phys. Res., Rio de Janeiro, Brazil). *J. Chromatography*, 1960, **3** (6), 584–585.—Develop on Whatman No. 1 paper for 18 hr. at room temp. with a mixture (1 : 1) of n-butanol and n-propanol previously shaken with a soln. containing 7 N $LiNO_3$ and 2 N HNO_3. R_F values are ^{210}Pb, 0·27; Te, 0·47; ^{210}Bi, 0·62; Se, 0·72; Po, 0·97.
　A. R. ROGERS

16. Thermolysis of sulphides precipitated by sodium sulphide. II. Sulphides of selenium, tellurium, gold, platinum, ruthenium, lead, bismuth, cadmium, silver, palladium and indium. I. K. Taimni and S. N. Tandon (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1960, **22** (6), 553–557 (in English).—The sulphides were examined with a thermobalance and the usual methods of drying and weighing were proved to be satisfactory, except for cadmium sulphide, which gave results 3% high when the ppt. was dried at the previously recommended temp. of 105° to 115°. A new factor of 0·75191 is proposed.
　R. M. S. HALL

17. Application of the RCA 1945 gauge to the analysis of hydrogen in metals. D. E. Swets (General Motors Corp., Michigan, U.S.A.). *Rev. Sci. Instrum.*, 1960, **31** (6), 659.—The sample is de-gassed in a vacuum furnace, and the evolved gas is expanded via gas pipettes into a known volume; the partial pressure of hydrogen in this expanded gas is measured with the RCA gauge (*cf.* Nelson, *Ibid.*, 1945, **16**, 273), and the initial hydrogen content of the sample can then be computed. Oxygen was found to have an effect on the calibration curve, but the effect of oil vapours from the pumps is minimised by the use of cold traps.
　G. SKIRROW

18. Determination of deuterium in water and organic compounds. Nobuo Tamiya (Medical and Dental Univ., Yushima, Bunkyo-ku, Tokyo, Japan). *Anal. Chem.*, 1960, **32** (6), 724–725.—Water is reduced to H by heating in a sealed tube for 3 hr. at 400° with platinum - zinc or platinum - zinc - asbestos catalysts (prep. described). The H is then examined in a mass spectrograph. Organic compounds are burnt in a combustion train, with platinum foil and silver gauze as catalysts, in the usual manner for a determination of H, and the water produced is frozen in a trap, and analysed as described above. The apparatus is illustrated.

R. E. E.

19. Determination of helium and neon in gases by chromatography. E. Zieliński (School of Mining and Metallurgy ‚Kraków, Poland). *Chem. Anal.*, *Warsaw*, 1960, **5** (2), 297–307.—The application of gas chromatography to the analysis of helium - neon mixtures was studied. The column efficiency was analysed as a function of the quality of certain Polish adsorbents, and an apparatus was constructed to permit satisfactory quant. analysis. Formulae are given and the results are tabulated and discussed. The mean error was $\pm 2 \cdot 0\%$.

L. SMAKOWSKI

20. Separation of the alkali-metal cations by electro-chromatography in paper. M. M. Tuckerman and H. H. Strain (Div. of Chem., Argonne National Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 695–698.—The mobilities of Na^+, K^+, Rb^+ and Cs^+ were measured in various organic solvents with paper as the stabilising medium. Good separations were achieved in 3 hr. on Eaton-Dikeman paper, Grade 301, previously washed with N HNO_3 and with H_2O; the electrolyte was 0·2 M ammonium formate and 0·4 M trichloroacetic acid in nitromethane; the potential gradient was 15 V per cm.

A. R. ROGERS

21. Paper chromatography in the separation of ions. Separation of alkali and alkaline-earth metals. A. K. Majumdar and B. K. Pal (Jadavpur Univ., Calcutta, India). *Z. anal. Chem.*, 1960, **174** (6), 429–431 (in English).—Ascending paper chromatography with the solvent systems 2-ethoxyethanol - H_2O - HCl (7:2:1) or acetone - H_2O - HCl (7:2:1) permits the quant. separation of alkaline-earth metals, including Be and Mg; Li, Na and K can be separated, but Rb and Cs travel with K. R_F values are tabulated.

J. P. STERN

22. Analysis of lithium metal and lithium (ionic) mixtures. M. Berkenblit and A. Reisman (Watson Lab., International Business Machines Corp., Columbia Univ., New York, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 721–722.—The procedure is based on the controlled hydrolysis of the metal in a stream of O, catalytic combination of the evolved H with the carrier gas, absorption of the H_2O in P_2O_5, and titration of the hydrolysis residue. The process is applied to the determination of Li in mineral-oil dispersions, and can be used for other alkali metals.

D. C. ARMSWORTH

23. The determination of lithium and its separation from other alkali metals. E. Blasius and F. Wolf (Hardenbergstr. 34, Berlin-Charlottenburg, Germany). *Z. anal. Chem.*, 1960, **174** (5), 349–351 (in German).—Mixtures of LiCl and NaCl are separated by extracting the LiCl with anhyd. dioxan in a reflux apparatus fitted with a P_2O_5 guard-tube and purged with dry nitrogen. Metallic sodium and calcium are added to the flask containing the dioxan to ensure the destruction of traces of water. Macro amounts are separated in 3 to 4 hr., micro amounts in ≃24 hr.

W. T. CARTER

24. Indirect complexometric determination of potassium. A. de Sousa (Anal. Chem. Lab., Fac. Sci., Lisbon, Portugal). *Anal. Chim. Acta*, 1960, **22** (6), 522–523 (in French).—Potassium is pptd. as perchlorate and this is reduced to the chloride, which is pptd. as AgCl. The ppt. is dissolved in ammoniacal nickel tetracyanide soln., and the liberated Ni^{2+} are titrated complexometrically with EDTA (disodium salt), with murexide as indicator.

R. M. S. HALL

25. Radiometric determination of potassium. V. E. Bel'skii and O. K. Fomin. *Zavod. Lab.*, 1960, **26** (6), 707–709.—The number of impulses (of β-radiation from ^{40}K) per min. per 1% of K is not a constant, but depends on the nature of the sample. By use of a number of compounds of K, as pure salts, mixtures and in solution, it is shown experimentally that this number is inversely proportional to the effective value of Z/A, *i.e.*, of $\Sigma(Z/A)_i P_i/100$, where $(Z/A)_i$ is the ratio of the atomic no. to the atomic wt. of the *i*th element in the sample and P_i is the percentage content. No correction is necessary, however, when the effective values of Z/A for sample and standard are not significantly different.

G. S. SMITH

26. Spectrographic determination of potassium, calcium and sodium chlorides in fluxes. N. L. Fishkova and L. N. Komlev (Podolsk Secondary Non-Ferrous Metal Works). *Zavod. Lab.*, 1960, **26** (5), 566–567.—A filtered aliquot (50 ml) of a 10% soln. of the flux mixture is treated with 10 ml of conc. HCl, 5 g of NH_4Cl (a stabilising buffer) and 2 ml of 10% $Co(NO_3)_2$ soln. (internal standard) and then diluted to 100 ml. A spark spectrum is excited with the soln. in a fulgurator (Fishkova and Rysina, *Ibid.*, 1959, **25**, 1459), and K (5 to 45%), Na (5 to 39%) and Ca (10 to 36%) are determined by use of the line-pairs K I 4044·14 - Co I 3569·38 A, Na I 3302·99 - Co I 3044·00 A, and Ca I 3630·75 - Co I 3569·38 A.

G. S. SMITH

27. Colorimetric micro-determination of rubidium and caesium. M. Kobrová (Bäderforschungsinst., Prague II). *Naturwissenschaften*, 1960, **47** (12), 278–279 (in German).—The method is based on conversion of rubidium chloroplatinate and caesium chloroplatinate into platinic iodide and measurement of the extinction, at 496 mμ for Cs and K and at 500 mμ for Rb. To the sample soln. add KI and dil. HCl and measure the extinction after 30 min. It is possible to determine fractions of a microgram per ml.

B. HEINING

28. Analytical uses of chelones. XXVIII. Detection of copper(II) with 1,2-diaminocyclohexanetetra-acetic acid. F. Bermejo Martínez and M. Guerrero Abella (Univ., Santiago de Compostela, Spain). *Z. anal. Chem.*, 1960, **174** (6), 411–413 (in German). —1,2-Diaminocyclohexanetetra-acetic acid (disodium salt) gives a bright blue complex with Cu^{2+} in aq. soln. at a pH between 4·5 and 12. In 10 ml of soln. 60 μg of Cu^{2+} is readily detectable and the limit of sensitivity is ≃25 μg. Interference by Ni^{2+}, Fe^{3+} and VO^{2+} is severe; colourless ions do not interfere.

J. P. STERN

29. Tritium in radiometric titrations: the copper-anthranilic acid system. G. H. Aylward, J. L. Garnett, J. W. Hayes and S. W. Law (Univ. of New South Wales, Sydney, Australia). *Chem. & Ind.*, 1960, (20), 560–561.—The results of the radiometric and amperometric titrations of Cu (10 mg) with ^{3}H-labelled anthranilic acid in 50 ml of acetate-acetic acid buffer (pH 4·6) indicate that the two end-points are almost identical. The two titrations were made simultaneously on the same sample, the Wilzbach procedure (*J. Amer. Chem. Soc.*, 1957, **79**, 1013) being used for the radiometric titration. The effect of the base-line error is much less than that of the ^{3}H measurement (1 to 2%).

W. J. BAKER

30. Simultaneous determination of dichromate and cupric ions. B. K. Sadananda Rao and G. S. Laddha (Alagappa Chettiar Coll. of Technol., Univ. of Madras, Guindy, India). *Z. anal. Chem.*, 1960, **174** (5), 324–328 (in English).—Dichromate and copper can be successively titrated with $TiCl_3$, with diphenylamine and $(NH_4)_2SO_4.FeSO_4$ - KSCN, respectively, as indicators. If the $TiCl_3$ soln. contains Fe^{3+} as impurity two standardisations are necessary, since the Fe^{3+} are active in the first titration but not in the second. *Procedure*—To the sample soln. containing $\simeq$100 mg of $Cr_2O_7^{2-}$ and $\simeq$100 mg of Cu^{2+} add H_2SO_4 (1:1) (15 to 20 ml) and dilute to 120 ml with water. Add a small amount of solid $NaHCO_3$ and titrate with 0·1 N $TiCl_3$, adding diphenylamine (0·005 M in conc. H_2SO_4) (0·2 ml) just before the end-point. Then add 10% KSCN soln. (10 ml), a few drops of 0·1 N $(NH_4)_2SO_4.FeSO_4$ and some solid $NaHCO_3$ and titrate with 0·1 N $TiCl_3$ until the colour changes from red to green. For 100-mg quantities of Cu^{2+} and $Cr_2O_7^{2-}$ the error is $\simeq \pm 0.5\%$.

W. T. CARTER

31. Controlled-potential electrolytic determination of copper in lead and tin-base alloys. B. Alfonsi (Res. and Control Lab., Auto-Avio, Fiat, Torino, Italy). *Anal. Chim. Acta*, 1960, **22** (5), 431–436.—Lingane's methods ("Electroanalytical Chemistry," Interscience Publishers, N.Y., 1953) have been extended for use with 5-g samples. For lead alloys, Ag and Bi are co-deposited with Cu and must be determined separately, and with concn. of Sb > 1·5% the deposit must be dissolved and re-electrolysed to avoid errors due to the co-deposition of Sb. Iron inhibits the deposition of Sb, but interferes with the determination of Cu. For tin alloys also, Ag and Bi are co-deposited with Cu; Pb and Cu can be determined successively. For lead alloys the accuracy is within 3% (12 determinations), and for tin alloys within 2% (15 determinations). Full instructions for the dissolution of the samples and the determinations are given.

R. M. S. HALL

32. Analysis of copper-refinery electrolyte by a condensed d.c. arc solution technique. N. Tomingas (Canadian Copper Refiners Ltd., Montreal East, Quebec, Canada). *Appl. Spectroscopy*, 1960, **14** (3), 72–75.—One drop of the sample is loaded on to the flat end of a graphite electrode (0·25 in. in diam.) and two such electrodes are arced together, with a Hilger-BNF type source unit at 210 μF, 15 ohms and 0·03 mH. Electrodes are rendered uniformly porous by being heated for 1 hr. at 540°. Cobalt chloride is added to the sample solutions as an internal standard for the determina-

tion of As, Sb, Ni, Fe and Bi in the range 0·1 to 10 g per litre with a coeff. of variation of $\simeq$3%.

P. T. BEALE

33. Spectrographic analysis of gold for purity. O. F. Degtyareva and M. F. Ostrovskaya. *Zavod. Lab.*, 1960, **26** (5), 564–566.—The flat end of a carbon rod is treated with a drop of aqua regia and ignited in a d.c. arc (20 to 30 amp.) for 30 to 45 sec. to remove Cu, Al and Fe. It is then coated with polystyrene lacquer (3·3% in benzene) and dried for 1 min. in air. A portion (0·04 ml) of the soln. of the sample prepared by dissolution in aqua regia and addition of an equal vol. of water, or of a standard soln. (containing 10% of Au), is placed on the treated end of the rod, which is then heated first at 80° to 90° and then at 160° to 170° to evaporate the soln. The spectrum is excited in an a.c. arc (6 amp.) and the elements Be, Cu, Mn, Ca, Mg, Cd, Al, Fe, Cr, Ba, Ni, K, Co, Pb, Zn and Ag are determined.

G. S. SMITH

34. Polarographic analysis of gold and gold alloy plating solutions. A. E. Knotowicz and G. Tatoian (The Patent Button Co., Waterbury, Conn., U.S.A.). *Plating*, 1960, **47** (6), 645–647.—The determination of Au, and of Ag, Cu, Ni and Zn as impurities or as components of cyanide plating soln., is described. For the determination of Ag and Au, use N KCN as supporting electrolyte, and read the diffusion current for Ag at -0.95 V and for Au between -0.95 and -1.7 V. Nickel gives a similar wave to Au so that a correction must be applied if Ni is present. For the determination of Cu, Ni and Zn, use N aq. NH_3 - N $(NH_4)_2SO_4$ as supporting electrolyte. Add 1 ml of conc. HNO_3 and 3 ml of conc. H_2SO_4 to the test soln. and heat to fuming. Cool, add gelatin soln. and dilute to 50 ml with the electrolyte, allowing the ppt. of Au to settle. Read the current for Cu at -0.8 V (correct for Ag), for Ni between -0.8 and -1.275 V, and for Zn between -1.275 and -1.6 V. Results for Au, Ag, Cu and Ni differ from chemical values by about 1%, for Zn by about 5%.

P. D. PARR-RICHARD

35. Rapid determination of gold in ores and rocks for prospecting purposes. V. Patrovský (Inst. Mineral Ores, Prague). *Geol. Průzkum*, 1959, (10), 302–303.—*Procedure*—To the finely powdered sample (0·5 to 10 g, containing 0·002 to 0·05% of Au) add conc. HCl (5 to 25 ml) and conc. HNO_3 (1 to 5 ml) and heat slowly on a water bath. Evaporate to a syrup and add HCl (1 ml) and H_2O (30 ml). Filter and wash the ppt. with H_2O ($\not> 20$ ml). To the filtrate add KBr (3 to 5 g) and extract with 15 ml and 10 ml of ethyl ether. Evaporate the combined extracts to dryness and treat the residue with HCl (1 ml) and bromine water (2 ml). Remove the Br by boiling, evaporate to dryness on a water bath and dissolve the residue in HCl (1:1) (0·5 ml). Dilute with H_2O (30 ml), add a satd. aq. soln. of salicylic acid (0·5 ml), conc. H_3PO_4 (0·2 ml) and 1% benzidine soln. in 20% acetic acid (1 ml), dilute with H_2O to 50 ml and measure the extinction with a violet filter. Compare with a calibration curve. Five samples can be analysed within 1·5 to 2 hr.

J. ZÝKA

36. The cupellation of gold-aluminium alloys. A. M. Scherhaufer (Hauptpunzierungs und Probieramt, Wien, Austria). *Z. anal. Chem.*, 1960, **174** (5), 352–354 (in German).—Gold-aluminium alloys containing 75% of Au which cannot be subjected to direct cupellation can be analysed by dissolving

the sample in 5 N NaOH, filtering off the residual gold and carrying out a normal cupellation. The method is considerably more rapid than the chemical separation of Au with H_2SO_3, and gives results agreeing to within 0·1%.	W. T. CARTER

37. [Spectrographic] determination of beryllium metal in air. R. J. Powell, P. J. Phennah and J. E. Still (Res. Lab., General Electric Co. Ltd., Wembley, England). *Analyst*, 1960, **85**, 347–353.—The sample is obtained by mechanical aspiration of air through filter-paper. The paper is wet-ashed with H_2SO_4 and a Br - HNO_3 mixture or, more rapidly, with a mixture of $HClO_4$ (0·5 ml), H_2SO_4 (1 ml) and Br - HNO_3 (5 ml). The residue is diluted, $Ca_3(PO_4)_2$ soln. is added followed by drops of aq. NH_3 until pptn. is complete. The ppt. is separated and washed by centrifuging, then dissolved in 0·2 ml of a soln. of $AuCl_3$ in HCl, the Au line 2913 A serving as an internal standard. The vol. of the soln. is adjusted to 1 ml and an aliquot (0·1 ml) is sparked in a porous graphite cup. The intensity of the Be line (3130 A) is measured, corrected for background and referred to a working graph prepared from standards ranging from 0 to 50 μg of Be per ml by the same analytical procedure. The method has been used only for samples consisting of metallic beryllium and the oxide produced by atmospheric oxidation. Modification of the chemical treatment might be necessary with beryllia refractories and other compounds of Be.	A. O. JONES

38. Spectrophotometric determination of beryllium in air and dust. R. H. A. Crawley (English Electric Co. Ltd., Atomic Power Division, Whetstone, England). *Anal. Chim. Acta*, 1960, **22** (5), 413–420. —Membrane air filters, types AF400 and AF600, have been shown to be much more efficient in trapping beryllium dust than No. 41 filter-papers, and ignition of the filter is also quicker. The ashed filter is fused with $KHSO_4$, the melt is extracted with dil. HCl, and any silica is removed by centrifuging. EDTA (disodium salt) is added to mask interference from <2 mg of Ca, Mg, Zn, Al, Ti, Co, Cu, V and Ni, and nitrilotriacetic acid to eliminate interference from Zr and reduce that due to Cr. The pH of the soln. is adjusted to between 4 and 7 with dil. aq. NH_3, a buffered soln. of aluminon (pH 5·8) is added, and, after making up the soln. to an appropriate vol. and heating on a water bath to complete the colour development, the extinction is measured at 530 mμ. If Fe is present, a correction must be made. Results are referred to a calibration graph covering the range 0 to 8 μg of Be. The lower limit of detection is 0·1 μg of Be, and recoveries on 0·5 to 5·0 μg range from 90 to 105%.	R. M. S. HALL

39. The use of hexa-amminocobalt chloride labelled with cobalt-60 for radiometric determinations. I. Determination of beryllium and bismuth. I. Dema, I. Gainar and T. Nascuţiu. *Rev. Chim., Bucharest*, 1960, **11** (5), 291–293.—The sensitivity of the method of Pirtea for Be (*Anal. Abstr.*, 1957, **4**, 2900) has been increased to 5 μg. For Bi the method of Pop (*Acad. R.P.R., Stud. Cercet. Chim.*, 1954, **2**, 245), based on the pptn. of $[BiCl_6][Co(NH_3)_6]$, is used. *Standardisation of reagent*—A 2% soln. of $[^{60}Co(NH_3)_6]Cl_3$ (**I**) (0·1 ml, activity $\simeq 10^4$ counts per mg of Co) is placed on a filter-paper disc, superimposed on an aluminium disc of the same thickness in a special Plexiglas filter-crucible (illustrated). The paper is dried (i.r. lamp) and its activity measured in the above

units in a G. - M. counter. *Determination of Be*— The faintly acid test soln. (1 ml ≡ 0·005 to 0·5 mg of Be) is treated with a slight excess of solid ammonium carbonate until the ppt. at first formed re-dissolves. The soln. of **I** (0·5 to 1 ml in excess) is then added dropwise, with stirring and rubbing the sides of the beaker, and the mixture is set aside for 15 min. The ppt. is then filtered off in the special crucible, washed with 60% ethanol containing a few drops of a conc. soln. of inactive **I**, then washed with 96% ethanol. The crucible is washed externally with the 60% ethanol soln. to remove traces of active material, wiped dry with filter-paper, then dried, and the activity (counts per min.) measured as described above, with the same counter and geometrical conditions. Then Be (mg) = 0·3060 × activity of ppt./activity of **I** soln. The maximum error for the range 0·007 to 0·35 mg of Be is −14·3%. *Determination of Bi*— The test soln. in 0·5 N HCl (1 ml ≡ 0·01 to 0·5 mg of Bi) is treated with **I** soln. as described above, and set aside for 1 to 2 hr. (overnight for 0·01 to 0·05 mg of Bi). The ppt. is then filtered off, washed with a soln. containing 2·5 ml of conc. HCl and 0·05 g of inactive **I** per 100 ml, and treated as described above; Bi (mg) = 3·546 × activity of ppt./ activity of **I** soln. The maximum error for the range 0·008 to 0·394 mg of Bi is −25%.	R. E. E.

40. Spectrographic determination of calcium and magnesium fluorides in aluminium electrolytic baths. I. L. Reznikov (Aluminium Works, Nadvoitsk). *Zavod. Lab.*, 1960, **26** (5), 570–574.—The soln. is placed in the hollow of an aluminium electrode and sparked with a flat-ended counter-electrode. Samples and standards should contain similar concn. of cryolite. The intensities of the lines Mg II 2798·06, Mg II 2790·79 and Ca II 3179·33 A are compared with Na I 3302·33/99 A. The method is applicable to contents of CaF_2 and MgF_2 from 1·5 to 10%. Carbon (>2%) interferes.

	G. S. SMITH

41. Colorimetric micro-methods for the determination of calcium based on the formation of coloured azo-derivatives of 8-hydroxyquinoline. V. Armeanu and P. Costinescu. *Rev. Chim., Bucharest*, 1960, **11** (6), 343–345.—The methods are based on the reaction of the Ca - 8-hydroxyquinoline complex with diazotised sulphanilic acid (**I**) or 7-amino-1-naphthol-3-sulphonic acid (**II**). *Separation of Ca*—The neutral test soln. (10 μg of Ca per ml) (5 to 10 ml) is warmed to 60°, treated with 2 to 5 ml of 2 N aq. NH_3 and 1 to 2 ml of 0·01 M ethanolic 8-hydroxyquinoline and heated gently till the ppt. becomes crystalline. The mixture is cooled to 40° to 50°; the ppt. is filtered off, washed with water till free from 8-hydroxyquinoline, then re-dissolved in 2 to 3 ml of warm 2 N HCl and diluted to 50 ml (soln. *A*). *Procedure with* **I**—The reagent is prepared by dissolving 0·1 g of **I** in 50 ml of ethanol, adding 10 ml of conc. HCl and diluting to 100 ml with water; to 20 ml is added 1 to 2 ml of 0·1 M $NaNO_2$ and the mixture is diluted to 100 ml. An aliquot (5 to 30 ml) of soln. *A* is treated with 5 ml of reagent followed by 5 ml of aq. NH_3 (1 : 4) and diluted to 50 ml with water. The extinction is read at once or after 24 hr. with or without a 480-mμ filter. The sensitivity is 1 in 4 × 10⁷ and the coeff. of variation is 0·09% with, and 0·11% without, the filter. *Procedure with* **II**—The reagent is prepared by dissolving 0·2392 g of **II** in 2 N Na_2CO_3 and diluting to 100 ml; 20 ml is treated with 3 ml of 0·1 M $NaNO_2$ and 2 ml of 2 N HCl and diluted to 100 ml.

An aliquot (5 to 40 ml) of soln. A is treated with 5 ml of reagent and 5 ml of NaOH soln. and diluted to 50 ml. The extinction is read at once or after 24 hr., with or without a 430-mμ filter. The sensitivity is 1 in $2 \cdot 3 \times 10^7$ and the coeff. of variation is $0 \cdot 11\%$ with, and $0 \cdot 12\%$ without, the filter.

R. E. E.

42. Micro-titration of calcium with EDTA. Magnesium interference. J. C. van Schouwenburg (Lab. of Soils and Fertilisers, Agric. State Univ., Wageningen, Netherlands). *Anal. Chem.*, 1960, **32** (6), 709–711.—The low results caused by Mg in the EDTA titration of Ca with murexide is probably due to adsorption of the Ca - murexide complex on the pptd. Mg(OH)$_2$. This is eliminated by the addition of Carbocel (Na carboxymethylcellulose) (**I**). *Procedure*—To the neutral test soln. (1 to 6 ml) add 25% (v/v) aq. triethanolamine soln. ($0 \cdot 2$ ml), $3 \cdot 2\%$ aq. KCN soln. [containing 3 ml of 10^{-3} M EDTA (**II**) per 100 ml] (2 drops), $0 \cdot 5\%$ aq. **I** soln. (containing 5 ml of **II** per 100 ml) ($0 \cdot 2$ ml) and N NaOH ($0 \cdot 4$ ml), stirring after each addition. Set aside for 30 min., then add fresh $0 \cdot 025\%$ aq. murexide (Merck) soln. ($0 \cdot 1$ ml), dilute the mixture to 8 ml and titrate with **II**. Phosphate does not interfere; Mg causes a slight positive error, and Zn and Mn may interfere if the Ca is not in comparatively large excess.

R. E. E.

43. Complexometric determination of calcium in calcium molybdate. N. M. Rakhmilevich. *Zavod. Lab.*, 1960, **26** (6), 717.—The sample ($0 \cdot 5$ g) is dissolved in 25 ml of conc. HCl, an aliquot (50 ml) of the diluted soln. (250 ml) is diluted to 150 ml, 2 to 3 drops of a soln. prepared by dissolving $0 \cdot 25$ g of indigo carmine in 100 ml of 25% ethanol are added, and the soln. is treated with 20% NaOH soln. until a yellow colour appears. It is then titrated with $0 \cdot 05$ M EDTA, in the presence of $0 \cdot 1$ to $0 \cdot 15$ g of murexide, to the appearance of a violet colour.

G. S. SMITH

44. Complexometric titration of calcium in the presence of large amounts of aluminium [in fluorspar and other minerals]. M. A. Popov (Central Lab., Geol. Dept., Novosibirsk). *Zavod. Lab.*, 1960, **26** (5), 540–542.—The sample ($0 \cdot 25$ g), after treatment with 8 ml of 10% acetic acid soln. followed by washing with water to remove CO_3^{2-}, is dissolved in 20 ml of neutral 8% AlCl$_3$ soln. The filtered soln. is diluted to between 120 and 150 ml and treated with either 15 ml of water - glycerol (1:1) or 10 ml of 15% sucrose soln. After addition of 2 or 3 drops of $0 \cdot 2\%$ indigo carmine soln., the soln. is made alkaline with 20% NaOH soln. (3 to 5 ml in excess) and treated with 1 to 3 drops of 10% Na$_2$SO$_3$ soln. and 5 to 7 drops of Acid chrome dark blue (C.I. Mordant Blue 7) indicator soln., prepared by dissolving $0 \cdot 5$ g in 25 ml of aq. NH$_3$ (sp. gr. $0 \cdot 91$) and diluting with ethanol to 125 ml. The Ca is then titrated with $0 \cdot 04$ M EDTA (disodium salt).

G. S. SMITH

45. Flame-photometric determination of strontium in silicates. M. Fornaseri and L. Grandi (Ist. di Geochim., Univ. Roma, Italy). *Geochim. et Cosmoch. Acta*, 1960, **19** (3), 218–221.—The sample (1 g) is decomposed with HClO$_4$ (5 ml) and HF (20 ml), then evaporated to dryness, and the residue is dissolved in hot water; Ca soln. (1%) (10 ml) is added to compensate for the effect of Al in silicates low in Ca, the soln. is made up to 50 ml, and divided into two 25-ml aliquots. The first is diluted to

50 ml to give soln. A; the second is mixed with a soln. of Sr (25 p.p.m. of SrO) (10 ml) and made up to 50 ml to give soln. B; A and B contain, respectively, x and ($x + 5$) p.p.m. of SrO. The emissions are then measured at 461 mμ and corrected by deducting the mean of the emissions at 454 and 463 mμ. Then $x = 5E_1/(E_2 - E_1)$, where E_1 and E_2 are the corrected emissions for A and B, respectively. The coeff. of variation ranged from 2 to $3 \cdot 25\%$ for the range 100 to 1500 p.p.m. of Sr.

R. E. E.

46. Gravimetric determination of strontium oxide in Portland cement. C. L. Ford (A.S.T.M.), 1916 Race Street, Philadelphia, Pa., U.S.A.). *Bull. A.S.T.M.*, 1960, (245), 71–75.—A comparative study of four different methods of determining small amounts of acid-sol. strontium in cement shows that the most suitable and accurate gravimetric procedure is that based on extraction of Ca(NO$_3$)$_2$ into 80% HNO$_3$ at 80° to 82° from the dry mixed nitrates of Ca and Sr. The residual insol. Sr(NO$_3$)$_2$ is washed several times with 80% HNO$_3$ and dried to const. wt. at 130° to 140° for $\simeq 2$ hr. The SiO$_2$, mixed oxides and Mn are removed initially by standard procedures, and the ignited ppt. of the mixed oxalates (one pptn. only is made) is used for the separation of Ca and Sr. The rate of heating (6 to 8 min.), temp. and very rapid stirring during the first 5 min. are critical for a successful separation; stirring time should be from 40 to 60 min. according to the approx. content of SrO. The lower limit of determination is $\simeq 0 \cdot 05\%$ of SrO, and the values are usually about $0 \cdot 02\%$ lower than those obtained by flame photometry.

W. J. BAKER

47. Complexometric determination of cation mixtures. II. Complexometric titration of zinc and copper in the presence of murexide as indicator. C. Liteanu, G. Murgu and L. Marinescu (Univ. "Babes-Bolyai," Cluj, Romania). *Z. anal. Chem.*, 1960, **175** (1), 1–4 (in German).—To determine the sum of Cu and Zn, the sample containing $\simeq 60$ mg of Cu^{2+} and Zn^{2+} is adjusted to pH $\simeq 5$ with NaOH, 10 ml of M NH$_4$Cl is added and the ppt. is dissolved in aq. NH$_3$. An aliquot of this soln. (A) at pH 8 to 9 is titrated at room temp. with 10^{-2} M EDTA in the presence of indicator (1 part of murexide and 200 parts of NaCl) to a colour change from yellow to violet. Zinc is determined in another aliquot of soln. A. After dropwise addition of M KCN until the aliquot is decolorised, and then 1 ml in excess, 2% formaldehyde soln. is added, and the pptd. Zn(OH)$_2$ is dissolved in aq. NH$_3$. The pH is adjusted to 8 to 9, and the soln. is titrated with 10^{-2} M EDTA, with murexide as indicator, at room temp., to a colour change from yellow to violet. The method is applicable to the analysis of ores, alloys and electroplating soln.

B. B. BAUMINGER

48. Analysis of cyanide zinc baths. Rapid determination of metal and total cyanide. A. Salka (Nymanbolagen AB, Uppsala, Sweden). *Metal Finish.*, 1960, **58** (5), 59–60.—Dilute 10 ml of the soln. to 100 ml. To 10 ml of the diluted soln. add H$_2$O (100 ml), 20% NaOH soln. (5 ml) and diphenylcarbazide indicator. Titrate with $0 \cdot 1$ N AgNO$_3$ until the red colour changes to yellow; hence calculate the NaCN. Add N NH$_4$Cl (10 ml) and conc. aq. NH$_3$ soln. (5 ml) and adjust the pH to $8 \cdot 0$ to $8 \cdot 5$ with dil. HCl. Titrate with $0 \cdot 1$ M EDTA (disodium salt) to Eriochrome black T; hence calculate the Zn.

N. E.

49. Determination of small amounts of zinc in metallic cadmium. P. V. Marchenko (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1960, **26** (5), 532–535.—The sample (2 to 5 g) is dissolved in 20 to 50 ml of 4 N HCl and the soln. is evaporated to dryness. The residue is dissolved in water and the soln. (50 ml) in a separating-funnel is treated first with 10 ml of 4 N HCl and 18 ml of H_2SO_4 (1:1) and then gradually with 10 ml of methylene blue soln. (prepared by treating 250 ml of a 1% soln. with 1 ml of conc. H_2SO_4 and 2 ml of 4% NH_4SCN soln. and filtering after 1 to 2 days) and 20 ml of 7·6% NH_4SCN soln. The soln. is diluted to 100 ml and set aside for 30 to 40 min. and then shaken with 5 ml of toluene so that the ppt. appears interfacially. The aq. layer is rejected and the ppt. is washed with 1% NH_4SCN soln. (3 × 10 ml), then dissolved in 10 ml of 4 N HCl and re-pptd. without removal from the separating-funnel. The washed ppt. is dissolved in 1 ml of H_2SO_4 (1:3), 1 ml of 30% H_2O_2 is added, and the soln. is evaporated to fuming. From the slightly acid soln. Cu is removed by extraction with dithizone soln. and the soln. at pH 5·5 is treated with 10 ml of 50% $Na_2S_2O_3$ soln. followed by extraction of the Zn with dithizone soln. The extract is washed first with a soln. prepared by mixing 75 ml of 20% Na acetate soln. and 10 ml of 50% $Na_2S_2O_3$ soln., diluting to 460 ml and adding 40 ml of 10% HNO_3 soln., then with 50% $Na_2S_2O_3$ soln., and finally with water. The Zn is re-extracted with HCl (1:1000) and the soln. is treated with 10 ml of 20% Na acetate soln. The Zn is then extracted again with dithizone soln., the extract is washed with 0·04% $Na_2S_2O_3$ soln., and the extinction is measured. The method is sensitive to 0·5 μg of Zn in 5 ml of extract.   G. S. SMITH

50. Colorimetric determination of zinc and cadmium with 8-quinolinol. W. L. Medlin (Field Res. Lab., Magnolia Petroleum Co., Dallas, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 632–634.— *Determination of Zn*—The sample ($>$ 100 μg of Zn) is dissolved in 7% HCl and the pH is adjusted to 8·8 to 9·5 with aq. NH_3; the reagent (5 g of 8-hydroxy-quinoline in 100 ml of H_2O containing 15 ml of glacial acetic acid) is added and the yellow complex is extracted with $CHCl_3$ and measured spectrophotometrically at 400 mμ. The method is valuable for the determination of Zn in the presence of large amounts of Ca; for other applications it is much less sensitive than the zincon method. *Determination of Cd*—The same procedure is followed except that the pH is adjusted to 7·6 to 8·6. The method offers no particular advantages over existing procedures, as the presence of Ca is not a serious problem in the determination of Cd.
                                                                R. E. E.

51. New indirect volumetric method for the determination of cadmium. J. Goldstein. *Rev. Chim., Bucharest*, 1960, **11** (6), 334–336.—The proposed method is based on the pptn. of $CdK_2[Fe(CN)_6]$ (**I**), the pptn. of the excess of $K_4Fe(CN)_6$ (**II**) by a standard soln. of $AgNO_3$ and back-titration of the excess of $AgNO_3$ with a soln. of NH_4SCN, with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ as indicator. *Procedure*—A soln. of the sample in H_2SO_4 neutralised to methyl orange with 10% aq. NH_3 is treated with 15 to 25 g of $(NH_4)_2SO_4$ (6 to 10 g per 100 ml of soln.) and an excess of a standard soln. of **II**. The mixture, diluted to 250 ml, is stirred, the pptd. **I** is allowed to settle for a few minutes, and then filtered off on a dry paper. A 50-ml aliquot of the filtrate is treated with an excess of 0·1 M $AgNO_3$. The pptd. $Ag_4Fe(CN)_6$ is filtered off immediately and a 50-ml aliquot of the filtrate is titrated with 0·1 M NH_4SCN. The method can be used in the presence of the alkali and alkaline-earth metals, but ions that are pptd. by either $[Fe(CN)_6]^{4-}$ or Ag^+ interfere. The error is $> \pm 0·1\%$ on the macro scale and $\pm 0·5\%$ on the micro scale.   H. SHER

52. Gravimetric determination of mercury by precipitation with sodium tetraphenylborate. A. Heyrovský (Charles' Univ., Prague, Czechoslovakia). *Analyst*, 1960, **85**, 432–434.—To the nearly neutral or faintly acid sample soln. (5 to 100 mg of Hg^{2+} at a concn. of $\simeq 1$ mg per ml) are added a few ml of satd. Na acetate soln. and then an excess of aq. Na tetraphenylborate soln., drop by drop, with stirring. The ppt. is collected in a sintered glass crucible, washed with a satd. aq. soln. of diphenylmercury, dried in a desiccator and weighed. Cations that form insol. ppt. with Na tetraphenylborate (*e.g.*, Ag^+, Tl^+, K^+, Cs^+, Rb^+ and NH_4^+), anions that form insol. phenylmercury salts (*e.g.*, Cl^-, Br^-, I^- and SCN^-) and Hg^+ interfere. Interference by metal ions pptd. as hydroxide or basic salt at the pH used can be avoided by addition of a chelating agent (*e.g.*, EDTA). Quoted results indicate an accuracy within 1%.   A. O. JONES

53. Titration of mercury(II) with sodium tetraphenylborate. A. Heyrovský (Charles' Univ., Prague, Czechoslovakia). *Anal. Chim. Acta*, 1960, **22** (5), 405–408.—The titration reaction proceeds in two stages—(*i*) 0·25 mole of Na tetraphenylborate (**I**) reacts with 1 mole of Hg^{2+} to form a clear soln. of phenylmercury salt; then (*ii*) further addition of **I** causes pptn. of diphenylmercury, and a persistent turbidity is taken as the end-point. For titration, the sample soln. should contain 5 to 50 mg of Hg^{2+}, and not more than 1 mg per ml. The soln. can be neutral, or slightly acid (0·05 N in HNO_3 or $HClO_4$) or buffered with Na acetate. The use of EDTA in buffered soln. eliminates interference from Cu, Zn, Mg, Ca, Mn and Ni; Ba, Fe, Co, Pb, Al and Bi do not interfere in acid soln.; and there is no interference from NO_3^-, ClO_4^-, PO_4^{3-}, ClO_3^-, SO_4^{2-}, NO_2^- or AsO_4^{3-}. Any anion that gives an insoluble phenylmercury salt (Cl^-, Br^-, I^- or SCN^-) interferes, as do Hg^+, but K^+ in concn. <3 times that of Hg^{2+} can be tolerated. Silver interferes in concn. $>0·5$ that of Hg^{2+}. When tested with $Hg(NO_3)_2$ and $Hg(ClO_4)_2$, the method had an error of $<1\%$ (for 2 to 30 mg of Hg^{2+}).   R. M. S. HALL

54. Potentiometric determination of mercury(II) with ethylenediaminetetra-acetic acid. H. Khalifa and M. G. Allam (Anal. Chem. Dept., Cairo Univ., Giza, Egypt). *Anal. Chim. Acta*, 1960, **22** (5), 421–427.—Mercury (200 μg to 100 mg) alone is determined with EDTA (disodium salt) (0·05 to 0·005 M) at pH 8 to 10, with a silver amalgam *vs.* S.C.E. system for the potentiometric end-point. The optimum pH for 0·005 M soln. is 6; 200 μg of Hg^{2+} can then be determined. Aluminium, Fe, Ti, Be and the alkaline-earth metals can be complexed or pptd. by F^-, which do not interfere. With binary mixtures of Hg and Ba, back-titration of excess of EDTA at pH 8 can be used to determine Hg, and at pH 11 for the determination of both metals. With binary mixtures of Hg and Pb, Mn or Zn, the total metal content can be determined as described above, and the titration repeated with Hg masked by I^-. With binary mixtures of Hg^{2+} and Ni, Cd or Co, the Hg^{2+} are titrated with KI

soln. and the total metals are determined by back-titration with $Hg(NO_3)_2$. Results are accurate to within 0.5%.   R. M. S. HALL

55. Catalysts for cerium(IV) oxidimetry. Determination of mixtures of mercury(I) and mercury(II).
W. H. McCurdy, jun., and G. G. Guilbault (Univ. of Delaware, Newark, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 647–650.—A mixture of $AgNO_3$ and $Mn(NO_3)_2$ in $HClO_4$ (prep. described) is used as a catalyst in the oxidation of Hg^I to Hg^{II} with $Ce(SO_4)_2$. The test soln. (15 to 300 mg of Hg^I as perchlorate, nitrate or sulphate) is treated with a 50 to 60% excess of $Ce(SO_4)_2$ soln. (prep. described) and 1 ml of catalyst, and sufficient $HClO_4$ to make the soln. 2 N. The mixture is heated at 90° till the colour becomes cherry-red (3 to 5 min.) and then cooled rapidly in ice, and the excess of $Ce(SO_4)_2$ is back-titrated with $(NH_4)_2SO_4.FeSO_4$ soln., with ferroin as indicator; 10 to 600 mg of Hg^I can be determined in the presence of 1 g of Hg^{II} with a coeff. of variation of ±0.3%. Bivalent mercury in $HClO_4$ soln. can be reduced to Hg^I by shaking with metallic mercury, and then determined as described above. Interferences are discussed.   R. E. E.

56. Colorimetric estimation of aluminium with catechol violet.
A. Anton (Textile Fibres Dept., E. I. du Pont de Nemours and Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 725–726.—Dissolve 0.3864 g of catechol violet in H_2O (100 ml). Mix 77 ml of reagent-grade pyridine with glacial acetic acid (63 ml) to give a buffer of pH 5. *Procedure*—To the test soln. (≯1.5 mg of Al) add buffer (10 ml) and reagent soln. (2 ml). Dilute to 100 ml with H_2O and measure the extinction against a reagent blank at 615 mμ. Zinc, Mg and Mn do not interfere up to 50 p.p.m., but Fe, Cu, Bi and Sn in concn. >2 p.p.m. interfere.
D. C. ARMSWORTH

57. Complexometric determination of aluminium and lead ions, with variamine blue as indicator.
L. Erdey and L. Pólos (Inst. of General Chem., Tech. Univ., Budapest, Hungary). *Z. anal. Chem.*, 1960, **174** (5), 333–337 (in German).—An indirect method is described in which the indicator system variamine blue, $K_4Fe(CN)_6$ and $K_3Fe(CN)_6$ at pH 5 is used (*cf. Anal. Abstr.*, 1958, **5**, 2547). *Procedure*—To the sample soln., containing 6 to 60 mg of Al or 20 to 500 mg of Pb, add 20 to 100 ml of 0.05 M EDTA (disodium salt) and 10 ml of buffer soln. (95·93 g of Na acetate and 295 ml of M acetic acid, made up to 1 litre with water). Boil for 3 to 4 min., then cool. Add 0.2 g of indicator [50 g of NaCl, 0.2 g of variamine blue, 0.422 g of $K_4Fe(CN)_6.3H_2O$ and 0.082 g of $K_3Fe(CN)_6$] and titrate with 0.05 M $ZnSO_4$. The alkali and alkaline-earth metals do not interfere. The standard deviations for 11 determinations on 27 mg of Al and 207 mg of Pb were ±0.065% and ±0.165%, respectively.   W. T. CARTER

58. Flame-photometric determination of small quantities of sodium in metallic aluminium.
K. H. Neeb (Forschungslab. Siemens Schuckertwerke A.-G., Erlangen, Germany). *Z. anal. Chem.*, 1960, **174** (5), 328–333 (in German).—Concn. of Na in aluminium below 0.001% are difficult to determine by flame photometry because of the increasing background from Al and the negative effect of Al on the emission of Na. Most of the Al can be removed simply and without contamination by reaction with ethyl bromide in a quartz apparatus to form $Al_2(C_2H_5Br)_3$, which is distilled off *in vacuo*. The residue is dissolved in dil. HCl and analysed by flame photometry. For a 2-g sample the lower limit of detection is 0.00001% of Na and the relative error is ±10%. In the range 0.0001 to 0.001% the relative error is ±5%. Calcium can be determined in a similar manner with a detection limit of 0.0005% and a relative error of ±10%.
W. T. CARTER

59. Quantitative spectrographic determination of gallium in solution.
A. Újhidy. *Magyar Kém. Foly.*, 1960, **66** (4), 132–134.—The vibrating a.c. arc method of Schiebe and Rivas (*Angew. Chem.*, 1936, **49**, 443) and the rotating graphite disc method of Boyle *et al.* (*J. Lab. Clin. Med.*, 1949, **34**, 625) and Mosher *et al.* (*Anal. Chem.*, 1951, **23**, 1514) have been applied to the determination of Ga (0.02 to 0.5%) in HCl soln. obtained in the processing of brown coal. Bismuth was used as internal standard, with the line-pair Ga 2943 A - Bi 2938 A. It was found that results by the Schiebe and Rivas method had a mean coeff. of variation of ±10% as compared with ±4.8% for those by the rotating disc method. Full procedural details are given, and the purification of the graphite discs is described.   R. E. E.

60. Photometric determination of gallium and indium by means of quercetin.
I. P. Alimarin, A. P. Golovina and V. G. Torgov (M.V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1960, **26** (6), 709–711.—*Procedure for Ga*—The soln. containing between 2.5 and 20 μg of Ga is treated with 1 ml of a 0.1% soln. of quercetin in ethanol, 5.5 ml of ethanol or methanol and 5 ml of an acetate buffer soln. (pH 4), and diluted to 25 ml. The extinction at 455 mμ is measured after 15 to 20 min. *Procedure for In*—The soln. containing between 10 and 100 μg of In is treated with 1 ml of 0.1% quercetin soln., 13·75 ml of ethanol and 5 ml of an acetate buffer soln. (pH 5), and diluted to 25 ml. The extinction at 416 mμ is measured after 10 to 15 min. Interference in the determination of Ga and In is caused by Al, F, oxalate, citrate and tartrate, but not by Zn or Cd at the relative concn. of Ga to Zn of 1:50, Ga to Cd ≃1:30, In to Zn ≃1:10, and In to Cd ≃1:10.   G. S. SMITH

61. New gravimetric method for the determination of scandium.
T. I. Pirtea. *Rev. Chim., Bucharest*, 1960, **11** (6), 336–338.—The method is based on the reaction of Sc with mercaptobenzothiazole (**I**) (sodium salt) with the formation of a white insol. cryst. ppt. of $Sc(C_7H_4NS_2)_3OH$. The reagent should be freshly prepared by dissolving 1 to 10 g of **I** in water, filtering off any residue, treating with N NaOH, adusting to pH 8 to 8·5 and diluting with water so that the content of **I** is 15 to 20%. *Procedure*—The soln. of scandium nitrate, sulphate or chloride (pH 5 to 6) (5 to 10 ml) is treated dropwise at room temp. with 2 to 3 ml of reagent; pptn. is immediate, and the ppt. is filtered on a sintered glass crucible, washed with 1% aq. **I** soln., then 3 times with 2 to 3 ml of water, and dried for 30 min. at 110° to 115°. The determination can be carried out directly in the crucible by adding to it an excess of the reagent, followed by an aliquot of the sample soln. with stirring, filtering by gentle suction and washing and drying as described above. Micro-determinations can be similarly carried out with an Emich filter-stick. Errors range from +0.5% on 80 mg to ±3.4% on 20 mg.   H. SHER

62. Accuracy of photometric determinations of cerium as cerium(IV) - sulphato-complexes. G. Gottschalk and H. Bartsch (Osram G.m.b.H. Studiengesellschaft, Berlin-Charlottenburg, Germany). *Z. anal. Chem.*, 1960, **174** (6), 423–429.—The absorption max. of the Ce - sulphato-complex (I) in N H_2SO_4 is at 315 mμ. The suitability of the Hg lines at 366 and at 436 mμ for photometry of I (14 μg to 14 mg per 100 ml) is discussed. The H_2SO_4 concn. must be between 0·8 and 1·5 N; the excess of persulphate should be $\not>$ tenfold. The extinction is constant at $20° \pm 2°$ for >6 hr. Interference by Cu^{II}, Al, colourless alkali- and alkaline-earth-metal ions, Sc, Y, Zn, Cd, Zr, Hf, Th and Sn is negligible. In the presence of Sr, Ba and Pb, the pptd. sulphate must be removed. At 366 mμ, Co does not interfere, Ni, La and Ti interfere slightly, and Fe^{III}, Cr^{III}, V^V and Mn strongly. At 436 mμ, Co, Ni, Fe and Ti interfere. In the presence of PO_4^{3-}, results for Ce are low. Methods for eliminating interferences are given.   J. P. STERN

63. Radiochemical determination of cerium by liquid - liquid extraction. J. J. McCown and R. P. Larsen (Chem. Engng Div., Argonne National Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 597–599.—Cerium can be separated from all other important fission products, without any precipitations or determination of chemical yield, by the method described. The Ce^{4+} are extracted with di-(2-ethylhexyl) hydrogen phosphate from a HNO_3 - $KBrO_3$ mixture, the organic layer is washed with the same mixture and the Ce is then extracted from it with a HNO_3 - H_2O_2 mixture. Traces of Ru are then removed by fuming the sample to dryness with $HClO_4$ in the presence of added $RuCl_4$. The Ce is then extracted with dilute HCl and the acid soln. is either plated for β-counting or γ-counted in a well-type scintillation counter. The coeff. of variation (10 analyses) on an irradiated uranium alloy was 1·4%.   K. A. PROCTOR

64. Spectrographic determination of rare-earth elements. Sh. G. Melamed, S. M. Polyakov and M. G. Zemskova. *Zavod. Lab.*, 1960, **26** (5), 554–556.—The sample (one part) is mixed with Y_2O_3 (19 parts) and carbon powder (20 parts) or with $CaCO_3$ (19 parts) and carbon powder containing 0·025% of Sc (20 parts) and excited in a d.c. arc between carbon electrodes. The total wt. of sample is 10 mg. The spectrum obtained during complete evaporation of the sample (120 to 130 sec.) is measured. Line intensities of the rare-earth elements are compared with the intensities of the Y or Sc lines.   G. S. SMITH

65. Continuous electrophoretic separations of radioactive rare-earth mixtures. I. Separation of cerium-144, terbium-160 and thulium-170, and of cerium-144, europium-152 and ytterbium-169 in 0·05 M lactic acid. Z. Pučar and Z. Jakovac (Inst. Rudjer Bošković, Zagreb, Yugoslavia). *J. Chromatography*, 1960, **3** (5), 477–481 (in English).—Autoradiographs of continuous electrophoretic separations and of discontinuous two-dimensional electrochromatographic separations of the mixtures are presented. Comparison with the activities of the separated rare-earth elements shows that complete separation is achieved. In the electrochromatographic separations, Ce, Tb and Eu are strongly adsorbed on the filter-paper. Advantages of the continuous electrophoretic method are (a) that the slower components moving in the direction of the electric field are not contaminated by a

reversibly adsorbed fraction of the faster components and absolute separation is achieved; (b) that carrier-free preparations are possible from very dilute solutions; and (c) that the method is particularly useful for hazardous radioactive materials since it is easily made automatic.

S. M. MARSH

66. The determination of bound and free carbon dioxide. Micro-determination of mixed alkali carbonate and hydroxide. E. Schulek and Á. Endróiné. *Magyar Kém. Foly.*, 1960, **66** (6), 211–213.—The interference of atmospheric CO_2 in the determination of weak acids and of dilute soln. of strong acids and alkalis is eliminated by the use as a sealing liquid of pentane containing propane and butane. It is established that the fading of the pink colour of phenolphthalein in even very dilute alkaline soln. is partly due to the rupture of lactone-type linkages in the phenolphthalein molecule, and it is recommended that a little more indicator should be added just before the end-point. The carbonate content of dilute alkali hydroxide soln. (0·1 to 0·01 N) can be accurately determined by means of $Ba(OH)_2$.   R. E. E.

67. Scintillation counting of carbon-14. D. S. Jenkinson (Rothamsted Expt. Sta., Harpenden, Herts., England). *Nature*, 1960, **186**, 613–614.—The method is a development of previous work (Schram and Lombaert, *Anal. Chim. Acta*, 1957, **17**, 417; Greenfield, *Analyst*, 1958, **83**, 114; Steinberg, *Nature*, 1958, **182**, 740) and is based on the counting of pulses of light emitted by a plastic phosphor in contact with a soln. of the isotope. The method is suitable for handling large numbers of samples with sp. activity of 0·1 to 10 μC in vol. of 6 to 12 ml, and is not dependent on the chemical form of the carbon. Since it is usually necessary to add a dye to prevent loss of light from the test cell, the colour of the soln. being examined is not important.

G. S. ROBERTS

68. Potentiometric determination of carbon in non-ferrous metals. W. Fischer and H. Bastius (Forschungsinst. für Nichteisen-Metalle, Freiberg-Sachs, Germany). *Metall.*, 1960, **14** (5), 429–434.—The procedure of Fischer and Schmidt (*Anal. Abstr.*, 1957, **4**, 432) has been adapted to the determination of traces of carbon in copper, titanium, titanium alloys, vanadium, nickel and molybdenum. With titanium and titanium alloys a 5-fold excess of copper should be added as a flux.

T. R. ANDREW

69. Rapid analysis of silica in refractories and minerals. Improved method of sample preparation. C. B. Belcher and L. B. Skelton (Broken Hill Proprietary Co. Ltd., Central Research Laboratories, Shortland, N.S.W., Australia). *Anal. Chim. Acta*, 1960, **22** (6), 567–570 (in English).—Previous methods are reviewed, and a simple method for the dissolution of the sample before subsequent colorimetric determination is proposed. The finely ground sample is sintered with Na_2O_2 in a platinum crucible at $500° \pm 5°$ for 10 min. The mixture is extracted with water and then H_2SO_4. Excess of peroxide, which interferes, is removed with $KMnO_4$. In the soln. the Si is determined photometrically at 813 mμ by the reduction of silicomolybdic acid to the blue complex with $Na_2S_2O_5$ and 1-amino-2-naphthol-4-sulphonic acid. A calibration curve is necessary. Beer's law is obeyed for samples (50 mg) containing 8 to 34% of Si. Interfering

PO_4^{3-} and Fe are masked with tartaric acid. Alumina, TiO_2 and Fe_2O_3 can be determined in the sample extract.　　　　　　　R. M. S. HALL

70. Rapid volumetric determination of silica in slags. B. Bieber and Z. Večeřa (State Inst. of Materials and Technol., Brno, Czechoslovakia). *Hutn. Listy*, 1960, **15** (5), 397–398.—To 0·2 g of finely powdered sample in a platinum dish add HNO_3 (1:1) (20 ml), heat till nitrous fumes disappear, then add HNO_3 (1:1) (20 ml) and cool to 40° to 50°. Add NH_4F (5 g) and mix till the soln. is clear. Add KNO_3 (15 g) and filter-paper pulp and mix for 5 min. Filter off the pptd. K_2SiF_6 on a polyethylene filter and wash with satd. neutral KNO_3 soln. till free from acid. Transfer the ppt. to a flask containing 10 ml of neutral 40% $CaCl_2$ soln. and 200 to 250 ml of hot H_2O, mix, add Tashiro indicator (10 drops) and titrate with 0·2 N NaOH till the soln. is green. Deduct a blank. The procedure takes 15 to 20 min. and the error is $\not> \pm 1\%$.　　　　　　　J. ZÝKA

71. Detection of lead by selective paper chromatography. M. Ziegler (Anorg.-Chem. Inst., Univ. Göttingen, Germany). *Z. anal. Chem.*, 1960, **174** (5), 323–324 (in German).—Lead (20 μg) can be detected in the presence of Hg^{2+} (20 mg), Cu^{2+} (3 mg), Ag^+ (15 mg) and Bi^{3+} (0·6 mg) by transferring the sample soln. containing the metals as nitrates to a paper strip carrying a band of CdS and developing for 4 hr. with a solvent containing isopropyl alcohol (50 ml), H_2O (45 ml), acetic acid (1 ml) and ammonium acetate (5 g). Only Pb^{2+} pass through the CdS zone and are detected above it by spraying with $(NH_4)_2S$ soln.

　　　　　　　W. T. CARTER

72. Use of EDTA for analysis of the active masses of an accumulator and of red lead. N. A. Filippova and T. F. Dubrovskaya. *Zavod. Lab.*, 1960, **26** (6), 711–716.—*Determination of PbSO₄. Procedure (a): Na_2CO_3 method for all active masses*—The sample (0·5 g for discharged masses, and 2 to 2·5 g for charged active masses) is boiled with 100 ml of 10% Na_2CO_3 soln. for 30 min. The soln. is filtered, the insol. matter is washed with hot Na_2CO_3 soln., and then with hot water, and the filtrate, after acidification to methyl orange, is treated with 3 ml of HCl (1:1), then boiled, and treated with 10 to 15 ml of hot 10% $BaCl_2$ soln. After 1 to 1·5 hr. at 50° to 60° the ppt. is filtered off and washed with hot water until Cl^- are removed. The paper and ppt. are boiled with 40 ml of 0·1 N EDTA (disodium salt), 30 ml of 25% aq. NH_3 and 40 to 50 ml of water, with the addition of aq. NH_3 as necessary, until the ppt. is dissolved. The cooled soln. is treated with 15 ml of an ammoniacal buffer soln., 10 ml of Mg - EDTA soln. [prepared by mixing 50 ml of a 0·1 N soln. of a salt of Mg and 15 to 20 ml of a buffer soln. (nature and concn. not stated) and diluting to 250 ml] and some Eriochrome black T - NaCl (1:100) indicator, and then titrated with $MgSO_4$ soln. to the colour change from blue to reddish-violet. *Procedure (b): acid method for negative masses*—The sample (0·5 g to 2·5 g) is treated with 15 to 20 ml of HCl (1:1) and 25 ml of water, and boiled with zinc filings. The transformation of $PbCl_2$ to Pb is complete when additional zinc filings remain bright for 2 to 3 min. The Pb is filtered off and washed. The filtrate is treated with 3 ml of HCl and the determination is completed as described above. *Determination of PbO_2 and total Pb in active masses*—The sample (0·2 g) is treated with 10 to

15 ml of 20% KI soln., 30 ml of 0·1 N EDTA (disodium salt) and 15 ml of 5% acetic acid soln. in a closed flask, and shaken until the PbO_2 is dissolved. The soln. is then titrated with 0·1 N $Na_2S_2O_3$ to give the content of PbO_2. The titrated soln. is mixed with 15 ml of 10% aq. NH_3, 15 ml of an ammoniacal buffer soln., 10 ml of Mg - EDTA soln. and some Eriochrome black T - NaCl (1:100) indicator, and titrated with 0·1 N $MgSO_4$ to give the content of Pb.　　　　　　　G. S. SMITH

73. Action of metavanadates on diphenylthiocarbazone [dithizone] and diphenylcarbazone. Application to determinations of lead in the presence of vanadates. J.-M. Bloch and J. Lazare (Lab. de Toxicol., rue de la Prairie, Nancy, France). *Bull. Soc. Chim. France*, 1960, (6), 1148–1150.—Both sodium meta- and hypo-vanadate form with dithizone a coloured vanadium complex [(dithizone)$_2VO_3$], which is soluble in H_2O and ethyl methyl ketone and thus interferes with the semi-micro determination of Pb when the ratio of V to Pb in the sample soln. is >2. Moreover, V and Pb are both present in the $CHCl_3$ extract of dithizone, and it is not possible to apply a correction for the content of V. A complete and convenient removal of V from the sample soln. can be effected with diphenylcarbazone. *Procedure*—Adjust the sample soln. ($\simeq 80$ ml, 20 to 200 μg of Pb) to pH 4, add 10% aq. Na citrate soln. (5 ml) and 10% aq. KCN soln. (2 ml), and again adjust the pH to exactly 3·8 with aq. 2·4% K H phthalate soln. (100 ml). Titrate the V with a 0·125% soln. of diphenylcarbazone in ethyl chloroacetate, withdrawing the organic layer after each addition (0·5 ml) of titrant until this increment no longer shows a rose colour. Adjust the pH of the V-free aq. layer to 9·5 (NH_4Cl - aq. NH_3 buffer) and determine the Pb by the usual dithizone procedure. The error for V is $\pm 5\%$.

　　　　　　　W. J. BAKER

74. Photometric determination of titanium with tiron. P. N. R. Nichols (Material Res. Lab., The Mullard Radio Valve Co. Ltd., Mitcham Junction, Surrey, England). *Analyst*, 1960, **85**, 452–453.—Certain disadvantages in the method of Yoe and Armstrong (*Anal. Chem.*, 1947, **19**, 100), in which tiron in a soln. buffered to pH 4·7 is used for the determination of Ti, appear to be due to the high pH value. The effect of lowering the pH was therefore examined, the extinctions after colour development being measured at 405 mμ. Results confirmed that a pH of 4·3 is necessary for max. colour development, but also showed a "plateau" between pH 2·3 and 4·0. The decrease in sensitivity ($\simeq 15\%$) is offset by the advantages of working at a lower pH ($3·0 \pm 0·2$). The use of a buffer is not necessary and the colour is stable for 24 hr. Beer's law is obeyed up to at least 80 μg of Ti per 100 ml, but the intensity of the colour is affected by change of temp. Any Fe^{3+} present up to 5 mg per 100 ml can be reduced with ascorbic acid or by warming with hydroxyammonium chloride soln., and pptn. of S which may accompany the use of sodium dithionite is thus avoided. A. O. JONES

75. Spectrographic determination of niobium and tantalum impurities in titanium. N. I. Tarasevich, K. A. Semenenko and N. F. Melëkhina. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 64–68.—A systematic study was made of the conditions of volatilisation and entry into the discharge zone of an a.c. and a d.c. arc of small amounts of Nb_2O_5 and Ta_2O_5 in the presence of large amounts of TiO_2.

As a result of this investigation the following method is proposed for the simultaneous determination of Nb (0·004 to 0·3%) and Ta (0·006 to 0·3%). Spectra are excited with a d.c. arc, at 12 amp. and 220 V. The electrode gap is 2 mm. The standards and samples ($\simeq 5$ mg), mixed with an equal wt. of carbon, are volatilised from the crater (3 mm deep) of the lower graphite electrode; the upper graphite electrode is cone-shaped (2 mm plane). Spectra are photographed. The line pairs used are Nb 2950·88 - Ti 2958·28 and Ta 2714·67 - Ti 2713·76 A. The calibration curves of log I_{Ta}/I_{Ti} and log I_{Nb}/I_{Ti} vs. log C are rectilinear after correction for background. The method is tested with artificial mixtures of TiO_2 with Nb_2O_5 and Ta_2O_5 and the results are tabulated. The coeff. of variation is $\simeq 8·2\%$ for Nb and Ta concn. of 0·004% and 0·006%, respectively.   K. R. C.

76. Determination of zirconium in titanium and its alloys by the catechol violet reaction. Yu. A. Chernikhov, B. M. Dobkina and E. I. Petrova. *Zavod. Lab.*, 1960, **26** (5), 529–531.—Improvements in the method previously described (Chernikhov *et al.*, *Anal. Abstr.*, 1960, **7**, 440) are given. By an increase in the concn. of EDTA and a reduction in the amount of reagent, 5 μg of Zr can be determined in the presence of 2·5 mg of Ti. With low concn. of Zr ($\not> 1$ μg per ml) the max. absorption occurs at 550 mμ (mol. extinction coeff. 25,000) instead of at 620 mμ. In alloys of titanium, Zr can be determined directly after dissolution of the sample in HCl, and Mo, Al and Nb do not interfere. With amounts of Nb $\not< 100$ μg a similar amount should be added to the blank soln.   G. S. SMITH

77. Determination of hydrogen in oxygen-containing zirconium hydrides by the hydrogen-evolution method. M. E. Straumanis, C. S. Lin and W. J. James (Dept. of Metallurg. and Chem. Engng, Univ. of Missouri, Rolla, U.S.A.). *Z. anal. Chem.*, 1960, **174** (5), 345–348 (in English).—The sample is dissolved in 3 N HF, the volume of the evolved hydrogen is measured, and the residue of ZrO_2 is collected, washed, dried and weighed. The hydrogen content is then calculated from the expression—
$$Z = [V - 491·43(A - R)]/10,627,$$
where Z is the weight (g) of H in the sample, V is the vol. of H evolved, A is the sample weight and R is the weight of Zr in the sample as oxide. The hydrogen content of 100-mg samples containing 1 to 2 mg of H can be determined with a precision of $\simeq \pm 10\%$. The method is not applicable to samples into which hydrogen has diffused at low temperatures.
     W. T. CARTER

78. Determination of oxygen in zirconium and Zircaloys by emission spectrography in an argon atmosphere. J. Artaud and C. Berthelot (Section de Contr. Anal. des Matér., Centre d'Études Nucléaires, Saclay, S. et O., France). *Mém. Sci. Rev. Métall.*, 1960, **57** (5), 338–434.—The polished sample (0·1 to 0·2 g) is diluted with $\simeq$ five times its wt. of platinum and subjected to a d.c. arc discharge between graphite electrodes in an atmosphere of Ar at 400 torr; O is then liberated as CO. The O line 7771·9 A is used for the determination, the Ar line 7891·1 A being taken as the internal standard. Standardisation is effected by arcing known amounts of ZnO.   J. H. WATON

79. Liquid scintillation spectrometry for the analysis of zirconium-95 - niobium-95 mixtures and coincidence standardisation of these isotopes. J. D.

Ludwick (General Electric Co., Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 607–610.—Mixtures of ^{95}Zr and ^{95}Nb at concn. ratios ranging from 50:1 to 2:1 can be analysed by liquid scintillation spectrometry. The results obtained agree well with those by other methods, and no correction for self-absorption is required.   K. A. PROCTOR

80. Separations involving sulphides. XII. Separation of thorium or titanium from some elements that form thio-salts. I. K. Taimni and S. N. Tandon (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1960, **22** (5), 437–438.—Sodium sulphide and HCl are added to the test soln., the pptd. sulphide is filtered off and weighed, and the Ti or Th in the filtrate is determined as the oxide or 8-hydroxyquinolinate, respectively. By this procedure As, Sb, Te, Se, Mo, Hg, Au, Pt and Re can be separated from Th, with an accuracy within 0·75%, and Sb, Te, Se and Hg from Ti to within 0·3%.   R. M. S. HALL

81. Spectroscopic determination of nitrogen-15 by means of a step filter. H. Faust (Agric. Chem. Inst., "Friedrich Schiller" Univ., Jena). *Z. anal. Chem.*, 1960, **175** (1), 9–18 (in German).—A detailed procedure is given. The precision of the method for samples containing 0·36 to 10% of ^{15}N is within $\pm 5\%$. Results are compared with those of the mass-spectrometric method.   B. B. BAUMINGER

82. Analysis of nitrogen for small concentrations of carbon monoxide by mass spectrometry. G. P. Schacher, R. E. Rippere and J. A. Hill (Gen. Engng Lab., General Electric Co., Schenectady, New York, U.S.A.). *Appl. Spectroscopy*, 1960, **14** (3), 79–80.—Because of the coincidence of the major peaks of N_2 and CO at mass 28, the determination of CO in combustion gases becomes difficult when the content is below 5%. By the use of Schütze reagent (prepared from silica gel, I_2O_5 and H_2SO_4), the CO may be converted into CO_2 and can thus be readily detected at concn. of 0·1%. A more accurate determination of the CO_2 obtained by the conversion from CO may be made by freezing out water and CO_2 from the sample before passage through the reagent.   P. T. BEALE

83. Determination of nitrogen in boron nitride. J. D. Cosgrove and E. C. Shears (Borax Consolidated Ltd., Cox Lane, Chessington, Surrey, England). *Analyst*, 1960, **85**, 448–449.—The sample (0·2 g) is fused with LiOH in a silver thimble enclosed in a test-tube carrying an inlet tube for the carrier gas (N) and an outlet tube leading the evolved NH_3 into 2% H_3BO_3 soln. The test-tube is immersed in sand and is heated to 500° to 550°. After 30 min. the H_3BO_3 soln. is titrated to methyl orange indicator with 0·25 N HCl, and the titration is continued at 10-min. intervals until there is no increase of titre during 30 min. Alternatively, the sample is heated with 80% H_2SO_4 in a sealed Carius tube at 320° for 8 hr. The contents of the tube are washed into a distillation flask, 45% NaOH soln. is added to the diluted liquid and the evolved NH_3 is distilled into 4% H_3BO_3 soln., which is finally titrated with 0·25 N HCl. The titre is corrected by means of a blank determination. Hydrolysis by 80% H_2SO_4 is the better method, but fusion with LiOH has the advantage that N and B may be determined in the same 0·2-g sample.
     A. O. JONES

84. Application of pyramidon [amidopyrine] to detection of nitrous acid. W. Wawrzyczek (General Chem. Dept., School of Agric., Olsztyn, Poland). *Nature*, 1960, **186**, 883.—When a few drops of a 5% soln. of amidopyrine and a few drops of dil. acetic acid are added to soln. containing NO_2^- in concn. as low as 0·009 mg per ml, a violet colour is formed which is not extracted by ether, $CHCl_3$, amyl alcohol or CCl_4. The reaction, which may be accelerated by heating, is not affected by sulphuric, nitric, iodic, hydrochloric, phosphoric, boric, oxalic, citric, tartaric or hydrobromic acid or by some bi- and ter-valent metal ions. K. A. PROCTOR

85. Spectrophotometric determination of phosphate using lanthanum chloranilate. Kiyoko Hayashi, Taeko Danzuka and Keihei Ueno (Dojindo & Co. Ltd., Kumamoto, Japan). *Talanta*, 1960, **4** (4), 244–249 (in English).—Phosphate reacts with lanthanum chloranilate to form insoluble lanthanum phosphate and free chloranilic acid which is determined by direct spectrophotometry on the filtered soln. *Procedure*—To the sample soln., containing 0·15 to 5 mg of phosphate, add 0·03 M Na succinate (5 ml), 95% ethanol (25 ml) and lanthanum chloranilate (0·1 g). Dilute the mixture to 50 ml and shake mechanically for 10 min. Filter a portion of the soln. through paper and measure the extinction at 530 mμ against a reagent blank. Read off the phosphate concn. from a calibration curve. Almost all cations interfere and must be removed by ion exchange; NO_3^- and Cl^- in concn. up to 400 p.p.m. do not interfere; SO_4^{3-} interfere but the effect can be eliminated by adding 800 p.p.m. of SO_4^{3-} to the sample and standards; F^- interfere seriously. The procedure has a precision of $\simeq \pm 2\%$. W. T. CARTER

86. Isotope dilution procedure for the determination of the true phosphate content of rock phosphates. J. Rijkheer (Research Dept., African Explosives & Chem. Ind., Ltd., P.O. Northrand, Transvaal). *J. S. Afr. Chem. Inst.*, 1960, **13** (1), 1–6.—In the procedure described, accurately weighed samples of standard $Mg_2P_2O_7$ labelled with ^{32}P (prep. described) were digested with the rock samples in acid soln., the soln. was passed through a column of Amberlite IR-120 resin (H^+ form) and phosphate in the percolate was isolated as NH_4MgPO_4, and ignited to $Mg_2P_2O_7$. The specific activity of this material was then compared with the standard, and the phosphate content of the rock was calculated in the usual way. All the phosphates must be in the ortho form. To ensure high accuracy the experimental procedure described must be closely followed. A. M. SPRATT

87. Quantitative analysis of alkali polyphosphates. O. Pfrengle (Forsch.-lab. der Chem. Fabrik Budenheim Rudolf A. Oetker KG, Wiesbaden). *Fette, Seif., Anstrichmitt.*, 1960, **62** (5), 433–439.—Various methods of determining the mean chain-length of a polyphosphate glass are compared. These include the Samuelson titration value (*Svensk. Kem. Tidskr.*, 1944, **56**, 343), the Na_2O to P_2O_5 ratio, and a paper-chromatographic method. Diagrams of typical chromatograms are given. (28 references.) H. M.

88. Amperometric determination of arsenic and antimony. V. A. Zakharov, O. A. Songina and N. A. Dragavtseva (Kazakh State Univ.). *Zavod. Lab.*, 1960, **26** (5), 537–540.—Two aliquots (5 or 10 ml) of the sample soln. are neutralised with NaOH soln.

(or $NaHCO_3$) and treated with 0·5 to 1 g of $NaHCO_3$, followed by dilution to 25 or 30 ml. In one soln. the sum of As and Sb is determined by titration with 0·01 N iodine at $+0·2$ V *vs.* the S.C.E. with a rotating platinum electrode. The other soln. is treated with an amount of 0·01 N K_2CrO_4 equivalent to 1·5 times the amount of iodine soln. needed in the first titration, and the As is titrated with 0·01 N iodine after $\geqslant 30$ sec. G. S. SMITH

89. Complexometric determination of arsenates. R. Vasiliev and G. Anastasescu. *Rev. Chim., Bucharest*, 1960, **11** (5), 298.—The proposed method is based on an indirect determination by means of Bi^{3+} in acid medium. *Procedure*—A soln. of the sample (0·3 g) in a few ml of water (containing 0·5 to 1 ml of 10% HNO_3 for water-insoluble samples) is treated with 40 ml of 0·05 M $Bi(NO_3)_3$ and diluted to 100 ml. The mixture is filtered; the first 20 to 30 ml is rejected, and the next 50 ml is treated in a titration flask with 0·5 ml of a 0·1% soln. of catechol violet as indicator and titrated with 0·05 M EDTA (disodium salt) until the first colour-change to violet. After the addition of 200 ml of water, titration is continued until the colour changes to yellow; NH_4^+, Li^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Al^{3+}, Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Cu^{2+}, Pb^{2+}, UO_2^{2+} and Ag^+ do not interfere; Fe^{3+}, Ga^{3+}, In^{3+}, Zr^{4+}, Th^{4+} and Hg^{2+} do. Perchlorates do not interfere, but Cl^-, SO_4^{2-}, PO_4^{3-} and $Cr_2O_7^{2-}$ do. A satisfactory method of eliminating Cl is the addition of 10 ml of a 10% soln. of $AgNO_3$ before the addition of the $Bi(NO_3)_3$ soln. Similarly 10 ml of a 10% soln. of $Ba(NO_3)_2$ eliminates SO_4^{2-}. Errors do not exceed those normally found in complexometric methods. H. SHER

90. Determination of antimony and tin in type metals by means of X-ray spectrography. A. J. Rossouw. *S. Afr. Ind. Chem.*, 1960, **14** (3), 44–46.—The X-ray spectrograph has been used to determine the Sb (11 to 16%) and Sn (4 to 10%) contents of antimony - tin - lead alloys. Samples were chill-cast from the melt to fit the spectrograph holder; pure lead was used to obtain a background value, and a standard alloy was used to obtain a calibration curve. A determination may be completed in 5 min. and results are estimated to be accurate to $\pm 0·2\%$. T. R. ANDREW

91. New method for the colorimetric determination of bismuth. E. Popper, V. Junie, L. Popa and L. Roman (Fac. of Pharm., Cluj). *Rev. Chim., Bucharest*, 1960, **11** (6), 341–343.—The proposed method is based on the pptn. of Bi^{3+} with 5-anilino-2-mercapto-1,3,4-thiadiazole (**I**), dissolution of the ppt. in an excess of **I**, and intensification of the colour by the addition of KI. Standards are prepared from a soln. containing 0·1256 g of Bi in 100 ml of 5 N HCl; aliquots of 0·005 to 2 ml are treated with 6 ml of fresh 1% ethanolic **I** soln. and 2 ml of 25% aq. KI soln., and diluted to 10 ml with 96% ethanol. The colours are read after 1 min. in a Lange photometer model N1 and the results are plotted. For larger quantities a soln. containing 2·5117 g of Bi per 100 ml is used, since pptn. occurs if more than 2 ml of test soln. is taken. Two curves are prepared covering the ranges 6 to 480 μg and 0·95 to 50 mg, respectively. Unknown samples are treated in the same way. Errors range from $+3\%$ to $-0·2\%$. The sensitivity is 6 μg per ml. It is claimed that the method can be adapted for the determination of Bi in the presence of other heavy metals; NO_2^- and oxidising ions interfere. H. SHER

92. Amperometric complexometric determination of bismuth in easily fusible alloys [containing cadmium, tin and lead]. Yu. I. Usatenko and M. A. Vitkina (F. E. Dzerzhinskiĭ Chem.-Tech. Inst., Dniepropetrovsk). *Zavod. Lab.*, 1960, **26** (5), 542–543.—The sample (0·5 to 0·6 g) is dissolved by boiling with 5 to 6 ml of HNO_3 (1:1), the soln. is treated with 4 to 5 ml of 0·5 M H_2SO_4 and evaporated to incipient fuming; 6 to 7 ml of HNO_3 (1:1) is added, the soln. is boiled for 1 to 2 min., three times its vol. of water is added, the soln. is again boiled, and the cooled soln. is diluted to 250 ml. An aliquot (25 ml) is treated with 3 g of KNO_3 and the soln. is titrated with 0·05 M EDTA (disodium salt) at +0·9 V *vs.* the S.C.E. with a platinum electrode.
G. S. SMITH

93. Colorimetric determination of bismuth and lead in copper and alloys. R. Socolovschi. *Rev. Chim., Bucharest*, 1960, **11** (6), 348–349.—The sample is dissolved in HNO_3, metals that give ppt. with aq. NH_3 and KCN are complexed with citrate, and Bi and Pb are extracted together with dithizone. They are then re-extracted into dil. HNO_3 and determined successively with dithizone; Tl^+, which interfere, are usually absent, and Sn^{2+}, which also interfere, are oxidised by the HNO_3. *Procedure*: (i) *extraction of Bi and Pb*—The sample (1 g) is dissolved in the minimum of conc. HNO_3 and filtered. Oxides of N are boiled off, the soln. is slightly diluted with water, treated with 40% ammonium citrate soln. (35 ml) and neutralised with conc. aq. NH_3; 20% KCN soln. is added slowly to the cooled soln. till complexing is complete, a further 1 ml of KCN soln. is added and the soln. is brought to pH 7·5 to 8 (phenol red). The mixture is then extracted with 0·003% dithizone soln. in $CHCl_3$ till the extract is colourless, and the Bi and Pb are re-extracted into 10 ml of N HNO_3. (ii) *Determination of Bi*—The HNO_3 extract is adjusted to pH 2 with aq. NH_3 and extracted with 0·001% dithizone soln. in $CHCl_3$ till the extract remains green. The combined extracts are then treated in a stoppered tube with more dithizone soln. in small portions till the colour becomes grey. A second tube containing the same amount of dithizone soln. is then titrated with standard Bi soln. (5 μg per ml in 0·01 N HNO_3) to the same grey colour. The error for $\simeq 0·001\%$ of Bi is ±10%. (iii) *Determination of Pb*—The HNO_3 extract after removal of Bi is neutralised to litmus paper with aq. NH_3 and treated with alkaline KCN - citrate soln. (50 ml of 5% KCN soln., 15 ml of 5% ammonium citrate soln., 5 ml of conc. aq. NH_3 and 450 ml of water) (20 ml). Dithizone soln. in $CHCl_3$ (0·003%) is added from a burette till a violet colour is obtained, and the excess of dithizone is removed by washing with alkaline KCN soln. (10 ml of 5% KCN soln., 5 ml of conc. aq. NH_3 and 500 ml of water) (2 × 30 ml). The org. layer is then suitably diluted with $CHCl_3$ and the extinction is read in a Pulfrich photometer with a green filter. The error for $\simeq 0·005\%$ of Pb is ±20%.
R. E. E.

94. Complexometric titrations (chelatometry). XLVII. Micro-determination of bismuth and lead by stepwise titration, with xylenol orange as indicator. Study of indicator colour changes with the use of photometric titration. S. Kotrlý and J. Vřešťál (Inst. Chem. Technol., Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1960, **25** (4), 1148–1164 (in German).—*Bismuth*—To a solution containing 0·004 to 0·08 mg of Bi (for photometric titration) or >0·1 mg of Bi (for visual titration) in 30 ml of $\simeq 0·1$ N HNO_3 add xylenol orange soln. and titrate with 0·001 M EDTA (disodium salt) photometrically at 530 mμ (measuring the decrease of extinction) or visually till the red colour changes to yellow. Chlorides and bromides interfere. *Lead*—To a soln. containing 0·004 to 0·08 mg of Pb (photometric titration) or >0·1 mg of Pb (visual titration) in 30 ml of the soln. at pH 5 (addition of hexamine) add xylenol orange and titrate with 0·001 M EDTA photometrically at 550 mμ or visually to a red - violet colour.
J. ZÝKA

95. Analysis of bismuth - antimony - tellurium - selenium combinations. J. F. Reed (Technol. Dept., Westinghouse Res. Lab., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 662–664.—Procedures are fully described for the successive determination of the elements named in a single sample. Selenium is pptd. as the free element with satd. aq. SO_2 soln. from a soln. of the sample in conc. HCl; the filtrate is diluted to 3 M in HCl. Tartaric acid is added to retain Sb in solution, and elementary Te is pptd. with SO_2 soln. and hydrazine hydrochloride soln. The filtrate is divided into two parts. In the first, Sb and Bi are pptd. with H_2S, the ppt. is fumed with H_2SO_4 and HNO_3, diluted with water, and reduced with SO_2 soln. The excess of SO_2 is boiled off, HCl is added, and the Sb is titrated potentiometrically with 0·1 or 0·05 N $KMnO_4$. The second portion is evaporated to dryness, the residue is re-dissolved in the minimum of N HNO_3, and diluted to 50 ml, and the Bi is titrated with 0·01 M EDTA (disodium salt) at pH 2·4 ±0·1, with thiourea as indicator. The accuracy and precision are within 2 parts per thousand.
R. E. E.

96. Precise method for determining vanadium in essentially pure vanadium metal. W. C. Dietrich (Union Carbide Nuclear Co., Y-12 Plant, Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1294, Mar., 1960. 10 pp.—Precision resulting in a limit of error (P = 0·95) for a single determination of ±0·079% is achieved by oxidising the weighed and dissolved sample to V^V and allowing this soln. to react with a weighed slight excess of $(NH_4)_2SO_4.FeSO_4.6H_2O$. The excess of reagent is then back-titrated with standard $K_2Cr_2O_7$ soln.
NUCL. SCI. ABSTR.

97. A new reducing agent. Reduction of vanadium(IV) and uranium(VI) with ferrous ion in catechol solutions. J. W. Miller (Res. Div., Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Talanta*, 1960, **4** (4), 292–293 (in English).—Ammoniacal soln. of V^{IV} and U^{VI} containing catechol are rapidly reduced by Fe^{2+} to V^{III} and U^{IV}, respectively. Potentiometric titrations may be carried out with a soln. containing M NH_4Cl, M aq. NH_3, 0·1 M Na_2SO_3 and 4% of catechol, with a mercury pool *vs.* a silver - silver chloride electrode; V^{IV} and U^{IV} cannot be differentiated and the V^{IV} must be removed before titrating the U^{VI}.
W. T. CARTER

98. Determination of tantalum and tungsten in rocks and meteorites by neutron activation analysis. D. H. F. Atkins and A. A. Smales (A.E.R.E., Harwell, Berks., England). *Anal. Chim. Acta*, 1960, **22** (5), 462–478.—After irradiation and dissolution of the sample and a standard in the presence of carriers, W and Ta are co-pptd. by tannin and cinchonine, and the ppt. is converted into the oxides and dissolved in $HF - H_2SO_4$. Tantalum is separated by solvent extraction with isopropyl ketone

or isobutyl methyl ketone, and finally converted into the tri-2,2'-dipyridyl ferrous fluorotantalate for counting; W is recovered from the aq. phase, and counted as the 8-hydroxyquinolinate. The activity is measured by either β- or γ-counting. The radio-chemical purity of the final source may be checked by half-life measurements, β-absorption curves and γ-spectra. Errors caused by alternative nuclear processes on W, Re and Os appear to be negligible. When standard samples were analysed the results were within $\pm10\%$ for Ta levels of 0.02% and within 0.05 to 0.2% for W. Results of duplicate analyses agreed to within $\pm10\%$. The limits of sensitivity attainable are 10^{-10} g for Ta and 10^{-11} g for W. Measurements have been made down to 8×10^{-10} g of Ta and 2×10^{-9} g of W with ^{182}Ta and ^{187}W.

R. M. S. HALL

99. Determination of ultra-micro amounts of sulphate as methylene blue. I. The colour reaction. L. Gustafsson (Dept. of Anal. Chem., Univ., Uppsala, Sweden). *Talanta*, 1960, **4** (4), 227–235 (in English).—Optimum conditions are outlined for the colorimetric determination of H_2S by conversion into methylene blue with p-aminodimethylaniline and Fe^{3+}. *Procedure*—Add the sample soln. (up to 70 ml) to a soln. 0.25 M in Zn acetate and 0.1 M in Na acetate (10 ml) and place in a thermostat at $20°$ for 10 min. Add p-aminodimethylaniline sulphate soln. (0.005 M in 3.5 M H_2SO_4) (10 ml), mix and add $(NH_4)_2SO_4.FeSO_4$ soln. (0.25 M in 0.5 M H_2SO_4) (2 ml). Dilute to 100 ml and measure the extinction at 667 mμ and $20°$ after 15 min. The reaction is sensitive to the acid concn. (optimum 0.3 M), but the concn. of the other reagents are not critical. Both the extinction and the yield are temperature-dependent, but once the colour is formed it is stable for several hours. Traces of Cu and Hg interfere seriously; de-ionised water must be used for all reagents and the Zn acetate soln. must be freed from heavy metals by pptn. with Na_2S. Beer's law is obeyed up to 25 μg of S, and the yield of methylene blue is $\simeq 65\%$ of the theoretical.

II. The reduction. L. Gustafsson. *Ibid.*, 1960, **4** (4), 236–243.—The sulphate is reduced to H_2S with NaH_2PO_2 and HI in a stream of N, and the H_2S is collected in Zn acetate soln. and determined colorimetrically as methylene blue, as described in Part I. *Procedure*—Place the solid sample containing up to 100 μg of S in a reduction apparatus (illustrated) consisting of a flask with a side arm, a reflux condenser and a delivery tube fitted with a gas washer containing water. Place the delivery tube in a flask containing water (68 ml) and Zn acetate - Na acetate soln. (10 ml). Add the reducing soln. [$NaH_2PO_2.H_2O$ (2.5 g), glacial acetic acid (25 ml) and HI (100 ml)] (3 ml) and pass N through the apparatus. Heat the contents of the flask rapidly to boiling and reflux for 10 min. Disconnect the flask and complete the determination as described in Part I. The presence of water in the sample causes low results and aq. samples must be evaporated to dryness before analysis. Nitrate ions and other oxidising agents interfere, but Fe^{III} (up to 20 mg), alkali metals and alkaline-earth metals do not. The standard deviation for a single result for 2 μg of S was ±0.05 μg (9 determinations).

W. T. CARTER

100. Determination of sulphate in chromium plating solutions. G. J. McKinley and A. Q. Jones (Anal. Control Group, Van der Horst Corp., Olean, N.Y.). *Plating*, 1960, **47** (6), 634–636.—By reduc-

tion of SO_4^{2-} to S^{2-} and titration with KIO_3 - KI reagent, $\ll 2$ g of SO_4^{2-} per litre is determined with an accuracy within $\pm1\%$; Fe and Cr up to 8 g per litre do not interfere. To prepare the reducing mixture, heat 200 ml of HI (sp. gr. 1.5 to 1.7) and 200 ml of HCl (sp. gr. 1.18) with 50 ml of H_3PO_2 soln. (50%) under reflux for 1 hr. under N. Store at $\not> 0°$. *Procedure*—Add 5 ml of test soln. to 30 ml of reducing mixture in a 100-ml flask, passing N until all iodine fumes are removed. Connect the flask to a vertical condenser, and the top of this to a delivery tube dipping into a flask which contains 150 ml of H_2O and 10 ml of $ZnSO_4$ soln. (50 g of $ZnSO_4.7H_2O$ in 250 ml of H_2O and 250 ml of conc. aq. NH_3; allow to settle and filter). Adjust the flow of N to 2 or 3 bubbles per sec. and heat the contents of the flask under reflux for 1 hr. Add starch soln. to the receiving flask and titrate with KIO_3 - KI soln. (0.7134 g of KIO_3 and 8 g of KI in H_2O to make 1 litre) to a permanent blue end-point.

P. D. PARR-RICHARD

101. [Spectrographic] determination of silicon and other impurities in selenium. V. L. Ginzburg and N. P. Glukhovetskaya (N.S. Kurnakov Inst. of Gen. and Inorg. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1960, **26** (5), 559–561.—Standards for silicon are obtained from a selenium alloy containing 3% of Si, which is first fused *in vacuo* at $750°$ to $800°$ for 3 hr., then mixed with pure Se to reduce the content of Si to 0.3% and fused *in vacuo* at $400°$. Further dilution is carried out similarly to give a range of standards. Samples and standards in cavities in carbon rods are excited in an a.c. arc. In the spectrum the line Se 2549.19 A is compared with one of the following lines of Si—2516.12 A for contents between 10^{-4} and 3×10^{-3} %, 2881.58 A for 2×10^{-4} to 2×10^{-2} %, 2514.33 A for 10^{-3} to 5×10^{-2} %, or 2435.16 A for 10^{-2} to 3×10^{-1} %. Determinations of Cu, Pb, Mg, Al, Ag, Fe, As, Sb, Ni, Bi, Te, Cd, Hg, Sn and Ni are carried out by means of standards, obtained by dilution with Se of an alloy of known composition containing larger amounts of these impurities.

G. S. SMITH

102. Separation of tellurium and iodine in different oxidation states by oxidation - reduction resins. R. Münze (Zentralinst. Kernphysik, Dresden, Germany). *J. prakt. Chem.*, 1959, **7**, 262–267.—When a TeO_3^{2-} soln. was added to a cation-exchange resin (Sn^{2+} form) the Te was retained on the column as the element. The reduction capacity was 9 mg of Te^{IV} per g of resin. The Te was not eluted with H^+ in the pH range 1.1 to 6.1, but was with HCl of concn. $> N$. The behaviour of aq. soln. of iodine, I^- and IO_3^- on the Te - Sn^{2+} column showed that the iodine species could be eluted free from Te. The continuous separation of ^{132}I species from the β-emitting ^{132}Te reduced on the Sn^{2+} column should be possible by this method. The IO_3^- were not reduced.

CHEM. ABSTR.

103. Determination of traces of lead in telluric acid. C. R. Veale and R. G. Wood (D.S.I.R., National Chem. Lab., Teddington, Middx., England). *Analyst*, 1960, **85**, 371–374.—For the preparation of the standard graph, 0 to 2.5-ml portions of standard lead soln. (4.17 μg of Pb per ml) are extracted by a modification of the procedure described by Sandell ("Colorimetric Determination of Traces of Metals," 3rd Ed., Interscience Publishers Inc., New York, 1959, p. 555) for isolating Pb. The soln. in HNO_3 is treated with an aq.

NH_2 - cyanide - sulphite soln. (*loc. cit.*) and 25 ml of a soln. of dithizone in CCl_4. The extinction of the filtered CCl_4 layer is measured at 520 mμ. A suitable wt. of the sample (up to 15 g) is dissolved in water, and HCl is added to adjust the pH to 1·6 to 2·0 according to the wt. of sample. The liquid is heated to b.p. after addition of hydrazine hydrate soln. and is set aside for <3 hr. The pH should then be 1·6 to 2·1. The supernatant liquid is removed through an immersion filter, the ppt. is washed, dissolved in HNO_3 and treated as in the preparation of the standard graph. The extinction of the final soln. is measured at 520 mμ against a reagent blank prepared by treating 5 ml of conc. HNO_3 similarly.   A. O. JONES

104. Oxidations with potassium permanganate in the presence of fluoride. III. Determination of chromium(III) and vanadium(IV). I. M. Issa and M. Hamdy (Cairo and Assiut Univ., Egypt). *Z. anal. Chem.*, 1960, **174** (6), 418–422 (in English).— The Cr^{III} or V^{IV} is oxidised within 5 min. by excess of $KMnO_4$ in NaOH soln. (0·3 to 1·8 M). Acidification with H_2SO_4, addition of 2% NaF soln. and then of excess of standard Hg^I soln. and back-titration of the excess with $KMnO_4$ at 40° in the presence of Cu^{2+} permit the determination of the $KMnO_4$ consumed without interference by Cr^{VI} and V^V; 1·8 to 4·5 mg of Cr^{III} and 50 mg of V^{IV} can be determined with an accuracy within ±0·5%.
                                        J. P. STERN

105. Determination of micro amounts of phosphorus in metallic chromium. A. A. Fedorov and F. V. Linkova. *Zavod. Lab.*, 1960, **26** (5), 535–536. —The sample (1 g for contents between 0·002 and 0·01% and 2 × 1·25 g for contents between 0·0002 and 0·002%) is placed in a 1-litre beaker (or in two 1-litre beakers) and dissolved in 70 ml of HCl (1:1) satd. with Br, an excess of Br being maintained throughout the dissolution. The soln. is evaporated to a syrup and then treated with a purified 3% soln. of KOH (500 ml in excess after neutralisation of the acid), prepared by treating 1 litre with 20 ml of 4% $Ca(NO_3)_2.4H_2O$ soln., setting aside for 2 hr., adding a further 8 ml of 4% $Ca(NO_3)_2.4H_2O$ soln., and filtering after 20 min. The soln. is treated with 20 ml of 30% H_2O_2 soln. [purified by shaking with freshly pptd. $Mg(OH)_2$], boiled for 20 min., treated in the cold with 8 ml of 4% $Ca(NO_3)_2.4H_2O$ soln., and set aside for 2 hr. After addition of a further 2 ml of the $Ca(NO_3)_2$ soln. and setting aside for 20 min. the ppt. is collected and washed with the KOH soln. It is then dissolved in 50 ml of hot HNO_3 (1:9), the filter being washed with 30 to 40 ml of hot HNO_3 (1:3) and then with hot water. The soln. is evaporated to dryness and the residue is dissolved in 10 ml (or 2 × 5 ml) of conc. HNO_3 and 15 to 20 ml of water. After addition of 2 ml of 4% $KMnO_4$ soln. [purified by boiling 300 ml for 5 min. with 4 g of $Fe(NO_3)_3.9H_2O$ and filtering after 30 min.], the soln. is boiled to precipitate MnO_2, which is then dissolved with a few drops of 5% $NaNO_2$ soln. Oxides of N are boiled off and the PO_4^{3-} in the cooled and diluted soln. are determined colorimetrically by the molybdenum blue reaction.   G. S. SMITH

106. Complexometric determination of molybdenum. J. B. Headridge (Dept. of Chemistry, The Univ., Sheffield, England). *Analyst*, 1960, **85**, 379–381.—To the approx. neutral soln. of molybdate (≡ 1 to 11 mg of Mo), adjusted to ≃10 ml,

1 ml each of conc. HCl and a saturated soln. of hydrazine sulphate are added and the mixture is maintained at 95° for 5 min. To the cooled liquid is added 10 ml of EDTA soln. (≃0·93 g of the disodium salt per 250 ml) and the mixture is transferred to a sample holder for an E.E.L. titrator or other filter photometer or spectrophotometer adapted for photometric titration. A buffer soln. (5 ml of a 45·9% soln. of Na acetate trihydrate) and alizarin complexone indicator (1,2-dihydroxyanthraquinon-3-ylmethylamine-NN-diacetic acid) are added, and with the use of an Ilford No. 604 filter the galvanometer reading is set to ≃8 and the soln. is titrated with standard $ZnCl_2$ soln. The end-point is ascertained from the titration graph and corrected for a blank determination. The procedure described will require modification if metals other than alkali or alkaline-earth metals are present.   A. O. JONES

107. Cerimetric determination of molybdenum in high-chloride media using the molybdenum blue reaction. C. M. Callahan, S. C. Foti and J. R. Lai (U.S. Naval Radiological Defense Lab., San Francisco, Calif.). *Anal. Chem.*, 1960, **32** (6), 635–637.— The neutral test soln. is acidified with an equal vol. of 2 N HCl and passed through a Jones reductor (previously washed with N HCl) at a rate of 20 to 30 drops per min. The percolate and washings are received under the surface of the receiver soln., or allowed to drop through an atmosphere of N. The receiver soln. should contain at least a 16-fold excess of Na_2MoO_4, and be at a pH such that an equiv. amount of molybdenum blue is formed; 5·5 to 6 g of $Na_2MoO_4.2H_2O$ in 100 ml of N HCl is satisfactory for up to 4·5 milli-equiv. of Mo. The soln. is then slowly titrated with standard $Ce(SO_4)_2$ soln. to a colourless end-point. The method is slightly less precise than the permanganate method, and the titration time is longer, but the $Ce(SO_4)_2$ soln. is very stable, it can be used in media containing much chloride, when the permanganate method is inapplicable, and no indicator correction is required.   R. E. E.

108. Chromometric determination of sexavalent molybdenum in the presence of thiocyanates. A. I. Busev and Gÿn Li. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 73–75.—The potentiometric titration of Mo^{6+} in HCl soln. with $CrCl_2$ in the presence of KSCN is studied with the use of a platinum indicator electrode. Mixtures of $(NH_4)_2MoO_4$ soln. (≃0·00959 g of Mo) (10 ml) and 6·1 N HCl (70 ml) are treated with CO_2 for 25 min.; KSCN (1 g) is then added and CO_2 is passed for another 5 min. The results for the titration at room temp. are tabulated.   K. R. C.

109. Photometric determination of molybdenum in the presence of tungsten by means of sodium 5-phenyl-2-pyrazoline-1-dithioformate. A. I. Busev, V. M. Bÿr'ko and I. I. Grandberg. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 76–80.— The complex of Mo with Na 5-phenyl-2-pyrazoline-1-dithioformate (I) is very stable and Beer's law is obeyed in the concn. range 0 to 8 μg per ml. With max. absorption at 530 mμ, the mol. extinction coeff. of the Mo - I complex extracted with $CHCl_3$ is 3200 and the detectable minimum is 0·6 μg per ml. At 530 mμ, Fe^{3+}, Cu^{2+}, Mn^{2+} and V interfere; W does not interfere as it does not form a coloured complex with I.   K. R. C.

110. **Analytical chemistry of benzoin α-oxime complexes of molybdenum, tungsten and vanadium.** H. J. Hoenes and K. G. Stone (Michigan State Univ., East Lansing, U.S.A.). *Talanta*, 1960, **4** (4), 250–263 (in English).—Sexavalent molybdenum is pptd. by benzoin α-oxime as $MoO_2(C_{14}H_{12}O_2N)_2$ from acid soln. in the range from pH 2 to 5% of H_2SO_4 by volume. Interference from Cr^{III} and V^{IV} is prevented by reduction with $(NH_4)_2SO_4.FeSO_4$ and that from Fe^{II} and Fe^{III} by washing the ppt. with 1% H_2SO_4; W^{VI} interferes and must be absent. The complex can be weighed after drying at 105°. In the solvent extraction of small amounts of the complex into $CHCl_3$, interference from V^V and W^{VI} is prevented by the addition of KH_2PO_4 and $(NH_4)_2SO_4.FeSO_4$. Sexavalent tungsten forms the complex $WO_3(C_{14}H_{12}O_2N)_2$, but quant. pptn. can be achieved only in the presence of quantities of KF too critical for unknown samples. The solubility of the tungsten complex in $CHCl_3$ is too low for quant. extraction; V^V forms an immediate yellow complex at pH 3. This is impure and changes on long standing into a white complex which has the composition $VO_2(C_{14}H_{12}O_2N)$. This is always contaminated with excess of reagent and is of no analytical value. The yellow complex is readily sol. in $CHCl_3$ and V^V can be extracted with benzoin α-oxime in $CHCl_3$ at pH 2·2 and determined colorimetrically with 8-hydroxyquinoline; Bi^{III}, U^{VI} and Sb^{III} interfere slightly. The Cr^{VI} complex with benzoin α-oxime was unstable and of no value for gravimetric or solvent-extraction procedures.

W. T. CARTER

111. **Colour complexes of catechol with molybdate.** G. P. Haight, jun., and V. Paragamian (Swarthmore Coll., Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 642–644.—The reaction of catechol with Mo^{VI} in aq. systems was studied. Two complexes (2:1 and 1:1) are formed, the most suitable for analysis being the 2:1 complex, the formation of which is most complete in neutral soln., which is maintained by a $Na_2S_2O_5$ buffer; this also prevents oxidation of the catechol by air. The molar absorbances and formation constants for both complexes are given.

G. P. COOK

112. **The surface acidity of tungsten(VI) and molybdenum(VI) oxides.** M. L. Freedman (Refractory Metals Lab., General Electric Co., Cleveland, Ohio, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 637–639.—Potentiometric titration with NaOH at high ionic strength was used. The surface acidity and solubility characteristics of tungsten trioxide depended on crystalline imperfections. Molybdenum trioxide did not exhibit surface acidity and the limiting pH for its continuous dissolution depended only on particle size. The layer lattice structure of molybdenum trioxide prevents the complete surface shielding which is postulated for tungsten trioxide.

D. C. ARMSWORTH

113. **Analytical chemistry of uranium. I. The determination of uranium by luminescence.** V. F. Grigor'ev, V. F. Luk'yanov and E. P. Duderova. *Zhur. Anal. Khim.*, 1960, **15** (2), 184–190.—The method depends on the measurement of the luminescence of fused discs and beads, prepared under controlled conditions with a suitable flux (*e.g.*, NaF or fluoride - carbonate mixtures). The apparatus described includes an electric furnace with a detachable platinum case for preparing the beads, a tube with piston for proportioning the powdered flux,

an apparatus for granulating the flux, and a photo-electric fluorimeter. A rapid method for preparing the beads is described. For a specimen with uniform distribution of U, the reproducibility of the readings is ±1·5%. For discs prepared with fluoride - carbonate flux and pure uranyl salts, the reproducibility is ±2%, and with NaF as flux, 6 to 7%. Methods for determining U in ores without preliminary purification are described.

R. E. E.

114. **Rapid extraction and spectrophotometric determination of uranium with 2-thenoyltrifluoroacetone.** S. M. Khopkar and A. K. De (Dept. of Chem., Jadavpur Univ., Calcutta, India). *Analyst*, 1960, **85**, 376–379.—To an aq. soln. of the sample ($\approx$ 0·2 to 1·2 mg of U) is added 10 ml of an acetic acid - ammonium acetate buffer soln. (pH 6) and, if other cations (*e.g.*, Ag^+, Th^{4+}, Zr^{4+}, Cu^{2+} or Fe^{3+}) are present, 2 ml of 0·1 M EDTA (disodium salt), and the mixture is extracted with 10 ml of 2-thenoyltrifluoroacetone (**I**) ($\approx$ 0·15 M) in benzene. The extinction of the yellow uranyl - **I** soln., appropriately diluted with benzene, is measured at 430 mμ against a reagent blank soln. treated similarly. At this wavelength the molar extinction coeff. (calculated on the basis of U) is 1954 ± 40. Oxalate, citrate, tartrate and CO_3^{2-} interfere, but 100-mg amounts of Pb^{2+}, Hg^{2+} or Ba^{2+} can be tolerated. As little as 2 μg of U per ml can be detected.

A. O. JONES

115. **Spectrophotometric determination of uranium with 1-(2-pyridylazo)-2-naphthol.** Shozo Shibata (Gov. Ind. Res. Inst., Nagoya, Japan). *Anal. Chim. Acta*, 1960, **22** (5), 479–484.—The ammoniacal test soln. containing 2 to 100 μg of U is treated with 1-(2-pyridylazo)-2-naphthol (**I**) in the presence of NaCl or Na_2SO_4 and the U-containing ppt. is extracted with $CHCl_3$. The sodium salts are necessary for complete extraction of the complex into $CHCl_3$. The extinction is measured at 560 mμ, with a reagent blank as standard. The spectra and the effect of pH on the complex are discussed. Beer's law is obeyed for 1 to 10 p.p.m. of U. EDTA competes with **I** and more **I** must be added when EDTA is present. The standard deviations of extinctions 0·180, 0·488 and 0·804 are, respectively, ±0·0021, 0·0040 and 0·0075 (11 determinations); thorium causes a positive error of <4% when in 40-fold excess. Of 24 metals listed, only Al and Sn interfere seriously; CN^- mask interference from Cu, Ni and Hg. R. M. S. HALL

116. **Spectrophotometric determination of uranium with 3-(2-arsonophenylazo)-4,5-dihydroxy-naphthalene-2,7-disulphonic acid (trisodium salt) [arsenazo].** H. P. Holcomb and J. H. Yoe (Pratt Trace Analysis Lab., Dept. of Chem., Univ. of Virginia, Charlottesville). *Anal. Chem.*, 1960, **32** (6), 612–617.—Uranyl ions give a stable blue complex with arsenazo, with an absorption max. at 590 mμ and a sensitivity of 1 in 3×10^5. The U is separated from interfering ions by extraction with ethyl ether by a modification of the method of Yoe *et al.* (*Anal. Chem.*, 1953, **25**, 1200). An aliquot (5 ml) of the filtrate from this procedure is adjusted with HNO_3 to pH 1 to 2, boiled to remove CO_2, and then adjusted to a pH between 2·5 and 5·5. Arsenazo soln. ($2·1 \times 10^{-3}$ M) (2 ml) is added, followed by Clark and Lubs borate buffer (pH 8·6) (10 ml), the mixture is made up to 50 ml and the extinction is read at 596 mμ against a reagent blank and referred to a calibration curve. Beer's law is obeyed over

the range 0·5 to 10 p.p.m. of U, and the colour intensity is constant at temp. between 15° and 35°. The coeff. of variation is 1·3%.   R. E. E.

117. Polarographic determination of sexavalent uranium in the presence of excess of ferric iron. A. A. Verbeek, J. T. Moelwyn-Hughes and E. T. Verdier (Dept. of Chem., Univ. of Natal, Pietermaritzberg, S. Africa). *Anal. Chim. Acta*, 1960, **22** (6), 570–576 (in English).—Uranyl ions at concn. of 10^{-3} to 10^{-6} M are determined in the presence of $\gg$ a 400-fold excess of Fe^{3+}. The supporting electrolyte is NaF soln. ($\simeq 0·75$ M), but the concn. is not critical; the pH must be $>6·5$. Titanium interferes, but Cu^{2+} and Mn^{2+} do not; Pb^{2+} in concn. $>10^{-3}$ M should be removed to avoid pptn. with the electrolyte. The precision is within $\pm 4\%$ at the 95% confidence level.   R. M. S. HALL

118. Determination of the stoicheiometry of uranium dioxide. Polarographic determination of uranium(VI) in uranium dioxide. Hisashi Kubota (Anal. Chem. Div., Oak Ridge National Lab., Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 610–612.—Sexavalent U in uranium dioxide can be determined by dissolving the sample in hot H_3PO_4 in an inert atmosphere and then polarographing in a H_3PO_4-$HClO_4$ medium. The deviation from the stoicheiometric composition of UO_2 is calculated by reference to the O to U ratio of 2:1. The method is best applied to concn. of U in the range 5 to 50 μg per ml, and to samples that are nearly stoicheiometric; the composition of a sample having an O to U ratio of 2·03:1 or less can be determined to within 0·001 O atom.   K. A. PROCTOR

119. Determination of neptunium in uranium-fission-product mixtures. Initial extraction with isobutyl methyl ketone. W. J. Maeck, G. L. Booman, M. C. Elliott and J. E. Rein (Atomic Energy Div., Phillips Petroleum Co., Idaho Falls, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 605–607.—In the two-cycle extraction system described, Np is oxidised with $KMnO_4$ and quant. extracted into isobutyl methyl ketone as a nitrate complex from an acid-deficient aluminium nitrate salting soln. containing tetrapropylammonium nitrate. By shaking with $FeCl_3$ - hydroxyammonium chloride soln. the Np is simultaneously reduced and extracted from the organic phase and is then re-extracted into thenoyl-trifluoroacetone - xylene. The extraction of U and Pu is $<0·1$ and $<0·01\%$, respectively. Ruthenium and Zr are decontaminated by a factor greater than 1×10^4.   K. A. PROCTOR

120. Rapid method for the determination of fluoride in liquids. S. A. Bartkiewicz and J. W. Robinson (Esso Res. Lab., Esso Standard Oil Co., Baton Rouge, La., U.S.A.). *Anal. Chim. Acta*, 1960, **22** (5), 427–431.—The sample (15 g for 100 p.p.m. of F⁻) is introduced into a modified Beckman oxy-hydrogen burner connected to a Vicor furnace, and the products of combustion are scrubbed in a sintered glass scrubber containing glass beads. Any conventional method can be used to determine F⁻ in the scrubber, and the thorium nitrate procedure (Belcher *et al.*, *Anal. Abstr.*, 1954, **1**, 492) is satisfactory. Tests with compounds that are normally difficult to decompose gave recoveries of 89 to 95%. The method is quick (20 min. per analysis) and any size of sample can be burned. An examination of 1,2-dihydroxyanthraquinon-3-ylmethylamine-*NN*-diacetic acid as a colorimetric

agent for F⁻ showed results to be satisfactory for 0 to 10 p.p.m. of F⁻, but Al, Fe, Ni, V, B and Cu interfere at the 100 p.p.m. level.   R. M. S. HALL

121. Determination of chloride by cathodic stripping polarography. Application to solutions of uranyl sulphate. R. G. Ball, D. L. Manning and O. Menis (Oak Ridge National Lab., Tenn., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 621–623.—In the method described, which is more sensitive than conventional polarographic methods, chloride in the concn. range 0·2 to 2 μg per ml is deposited as Hg_2Cl_2 on a mercury-pool micro-electrode at $+0·4$ V *vs.* the S.C.E. for 15 min., the sample soln. being stirred magnetically during the deposition. By changing the potential to less positive values the deposit is stripped from the electrode, and the peak height of the recorded current - voltage curve is measured and related to the chloride concn. by means of a calibration curve. The coeff. of variation is $\simeq 5\%$.   K. A. PROCTOR

122. Argentimetric determination of chlorine in tetra-n-butyl titanate. Ya. I. Chulkov. *Zavod. Lab.*, 1960, **26** (5), 550.—The sample (0·3 to 1 g) is dissolved, at 110° to 140°, in a mixture of 15 ml of acetic anhydride and $\ll 4$ g of NH_4F. To dissolve the ppt., the soln. is heated at 50° to 55° with the minimum amount of water. The Cl⁻ in the cooled soln. are then determined by titration with $AgNO_3$ soln.   G. S. SMITH

123. The adsorption of some chloro-complexes on cellulose. T. J. Beckmann and M. Lederer (Inst. du Radium, Lab. Curie, Paris, France). *J. Chromatography*, 1960, **3** (5), 498–499 (in French).—Un-impregnated cellulosic paper has been shown to adsorb chloro and bromo anionic complexes from solutions in HCl or LiCl. Results for the chromatography of anions from Au^{III}, Ce^{III} and Fe^{III} in solutions of HCl and of LiCl containing 1% of 10 N HCl, by an ascending technique, are presented and show possible value for analysis.   S. M. MARSH

124. The simultaneous determination of bromide and iodide. A. de Sousa (Anal. Chem. Lab., Fac. Sci., Lisbon, Portugal). *Anal. Chim. Acta*, 1960, **22** (6), 520–522 (in French).—Two aliquots of sample soln. are treated with $AgNO_3$. The ppt. from one aliquot is oxidised with $K_2Cr_2O_7$ and H_2SO_4 to convert I⁻ into IO_3^- and liberate Br, which is removed in a current of air. The IO_3^- are then reduced to I⁻ with Na_2SO_3 and pptd. as AgI. This ppt. and the ppt. of combined halides from the second aliquot are dissolved in ammoniacal nickel tetracyanide, and the displaced Ni^{2+} are titrated with EDTA (disodium salt). Bromide is determined by difference.   R. M. S. HALL

125. Complexometric determination of bromides and bromates in the presence of each other. A. de Sousa (Chem. Lab., Fac. Sci., Univ. of Lisbon, Portugal). *Z. anal. Chem.*, 1960, **174** (5), 337–339 (in English).—The method described is based on that of Flaschka and Huditz (*Brit. Abstr. C*, 1953, 51). Actual bromide is pptd. as AgBr, which is dissolved in an ammoniacal soln. of $K_2Ni(CN)_4$, and the liberated Ni^{2+} are titrated with EDTA (disodium salt) soln., with murexide as indicator. Bromate, in another portion of sample, is reduced to bromide with arsenous oxide, and the total bromide is determined as described above. Chlorides, iodides and heavy metals interfere.   W. T. CARTER

126. Coulometric determination of small amounts of iodide by internal electrolysis. J. Kis and C. Schejtanow (Inst. f. Anorg. Chem., Tech. Univ., Budapest). *Period. Polytech.*, 1960, **4** (2), 163–170 (in German).—The determination of 30 to 100 μg of iodide can be carried out in $\simeq 30$ min. by internal electrolysis, with the hydrogen micro-coulometer described by Proszt and Poós (*Ibid.*, 1957, **1**, 25) to measure the amount of electricity which flows during the oxidation of iodide to iodine. The cathode consists of a lead dioxide - sulphuric acid paste contained in an extraction thimble impregnated with collodion; connection to it is made by a carbon rod. The anode is a cylinder of platinum gauze surrounding the cathode, and connected to it through the micro-coulometer. The basal soln. (2% aq. KNO_3) is stirred by a stream of N. The relative error of the determination is $<3\%$.

R. M. ROWLEY

127. Photometric determination of manganese. H. Hartkamp (Inst. f. Spektrochem. u. angew. Spektroskopie, Dortmund-Aplerbeck, Germany). *Angew. Chem.*, 1960, **72** (10), 349.—Pyridine-2,4-dicarboxaldoxime and pyridine-2,6-diacetaldoxime (**I**) at pH 9·5 to 11·5 are suitable for the photometric determination of Mn in the range 0·1 to 10 μg per ml. Soluble green complexes, probably containing Mn^{III}, and becoming red at high metal concn., are formed with Mn^{2+}, and obey Beer's law over the concn. range stated. The colour develops very rapidly after warming the test soln. for a short while with a small excess of reagent, and does not alter for several hours. Reducing agents, *e.g.*, ascorbic acid, hydroxyammonium chloride and hydrazine sulphate, interfere. Metals of groups I to IV are without influence, but Fe, Co and Ni must be absent. The complex with **I** has a max. extinction at 598 mμ.

J. H. WATON

128. Mass-spectrometric identification and determination of technetium. G. M. Kukavadze, R. N. Ivanov, V. P. Meshcheryakov, Yu. G. Sevast'yanov, B. S. Kir'yanov, V. I. Galkov and A. P. Smirnov-Averin. *Atomnaya Energiya*, 1960, **8** (4), 365–366.—Surface ionisation of the test substances on incandescent iridium-coated (0·1 μ) tungsten emitters provides the ion stream. Aq. ammonium pertechnetate soln. is transferred by micro-pipette to the emitter. Technetium is then reduced to the metallic state by heating the emitter to redness in a stream of H for a few minutes. The ion source with the treated sample is placed in the mass spectrometer, and Tc^+ are produced at 1600° to 1800°. Mass spectrograms of ^{99}Tc and of ^{87}Rb and ^{85}Rb are given; Rb provides a reference peak, and is present as an impurity in the iridium. The method described, in combination with isotope dilution or integral mass-spectrography, is satisfactory for the determination of Tc.

K. R. C.

129. Method for "ferrous iron" and "excess oxygen" in rocks, minerals and oxides. C. O. Ingamells (Mineral Constitution Lab., Pennsylvania State Univ., University Park, U.S.A.). *Talanta*, 1960, **4** (4), 268–273 (in English).—The method depends on the stability of both Mn^{III} and Mn^{II} in phosphoric acid - pyrophosphate mixtures. The sample is dissolved in a phosphoric acid mixture containing an excess of Mn^{II} with or without the addition of standard oxidant, and the Mn^{III} remaining in the soln., or produced by reaction with the sample, is titrated with $(NH_4)_2SO_4.FeSO_4$. *Procedure*—Mix 85% H_3PO_4 (20 ml), 50% $NaH_2PO_4.H_2O$

soln. (5 ml) and 0·1 N $K_2Cr_2O_7$ (0·5 ml) and heat without boiling until water is expelled and the excess of chromate is destroyed. Cool, add water (2 ml), 50% $MnSO_4.H_2O$ soln. (1 ml) and a measured excess of 0·1 N $KMnO_4$, evaporate off the excess of water and add the finely ground sample ($\div 1$ g). Heat until dissolution is complete, cool, dilute with water until the concn. of H_3PO_4 in the soln. is $\simeq 20\%$ and titrate with 0·01 N $(NH_4)_2SO_4.FeSO_4$, with Ba diphenylaminesulphonate as indicator. The results obtained agree well with those obtained by other methods, but the procedure is not applicable to samples that do not dissolve readily in the reagent, or that form peroxides in acid soln. or that contain sulphur or organic carbon; Co_3O_4, Pb_3O_4 and certain rare-earth-metal oxides evolve oxygen during dissolution and give low results for excess oxygen.

W. T. CARTER

130. Analytical aspects of some organic acids. VI. Direct complexometric titration of iron(III) and indirect EDTA titration of zirconium and thorium using o- and p-cresotic [2- and 6-hydroxy-m-toluic] acids as indicators. C. S. Pande and T. S. Srivastava (Lucknow Univ., India). *Z. anal. Chem.*, 1960, **175** (1), 29–34 (in English).—Macro (3 to 7·8 mg) and micro (0·16 to 2·6 mg) amounts of Fe^{3+} can be titrated directly with EDTA, with 2- or 6-hydroxy-m-toluic acid (**I**) as indicator. At the end-point the colour change is from blue - violet to pale yellow. *Procedure*—The test soln. containing 0·16 to 2·6 mg of Fe^{3+} is adjusted to pH 3·6 by adding acetate buffer and diluting with twice-distilled H_2O to 25 ml. After an addition of 0·05 g of solid **I** the soln. is titrated with 0·05 M EDTA (disodium salt) until the violet colour is discharged. There is no interference from Be, Mg, Ca, Sr, Ba, Cd, UO_2^{2+} or Mn^{2+}, but Cu^{2+}, Zn^{2+}, Pb^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, ZrO^{2+}, Al^{3+}, Cr^{3+}, Ti^{4+}, Ce^{4+} and Th^{4+} should be absent. The indirect EDTA titrations of Zr and Th, with **I** as indicator, have also been carried out. To the sample soln. is added 0·1 M EDTA (disodium salt) in slight excess, and the pH is adjusted to 6·0 by adding dil. aq. NH_3 and boiling the soln. gently for $\simeq 2$ min. The soln. is then cooled to 31°, the pH is adjusted to 4 or 3·6 and the soln. is diluted to $\simeq 100$ ml. Excess of EDTA is back-titrated with 0·1 M Fe^{3+}, with 0·1 g of **I** as indicator. Some typical results are given.

B. B. BAUMINGER

131. Low-pressure method for carbon analysis in steels: a note. V. Ramakrishna (Research & Control Lab., Tata Iron & Steel Co., Jamshedpur, India). *Anal. Chim. Acta*, 1960, **22** (6), 592–594 (in English).—The carbon dioxide cold-trap of Cook and Speight (*Analyst*, 1956, **81**, 144), when used with liquid-air coolant, froze out oxygen from the gas stream. A modified trap is proposed and described, together with a method for its use.

R. M. S. HALL

132. Determination of tin in pig iron and steel. C. Mahr and K. Waffenschmidt. *Arch. Eisenhüttenw.*, 1960, **31** (4), 221–225.—Four procedures are given for the determination of about 0·005 to 0·1% of Sn. Three of these, suitable only for samples containing <2% of Cr, rely on co-pptn. of the hydroxides of Sn and Al, or distillation of $SnCl_4$, or a combination of both techniques, followed by polarographic determination in a basal soln. 10^{-4} M in tetraphenylarsonium chloride and 6 M in HCl containing a few drops of cresol red. In the fourth procedure, the sample is dissolved in an oxidising

medium, an excess of iron powder is added, the mixture is heated under a Bunsen valve, and the reduced sample is titrated potentiometrically with KIO_3 soln. (0·005 N) after addition of KI.

T. R. ANDREW

133. Determination of nitrogen in steel. J. Llop Llop (Lab. de Altos Hornos de Cataluña, S.A.). *Inst. Hierro y Acero*, 1960, **13**, 532–535.—Some nitrogen is lost when steel is dissolved in HCl but not when H_2SO_4 is used. The sample of carbon or alloy steel (3·5 g) is dissolved in H_2SO_4 (1:4) (50 ml) in an atmosphere free from NH_3. After the addition of tartaric acid, the soln. is transferred to a steam-distillation apparatus (illustrated) and made alkaline. The NH_3 is distilled off and absorbed in 0·005 N H_2SO_4, the excess of acid being back-titrated with 0·025 N NaOH. A reagent blank is carried out, and the content of nitride nitrogen is calculated. Nitrides not decomposed by dil. H_2SO_4 are filtered off, fused with K_2SO_4 and added to the sample soln. The procedure takes 20 min.

G. H. FOXLEY

134. The accurate determination of cobalt. A. G. Foster and W. J. Williams (Royal Holloway College, Englefield Green, Surrey, England). *Anal. Chim. Acta*, 1960, **22** (6), 538–546 (in English).—A critical review is given of the possible primary standards for the determination of Co, and, of those investigated, $[Co(NH_3)_3Cl]Cl_2$, $[Co(NH_3)_6]Cl_3$ and $K_3[Co(CN)_6]$ are preferred. The errors are $<0·7\%$.

R. M. S. HALL

135. Complexometric estimation of micro amounts of cobalt using ethylenedinitrilotetra-acetic acid. R. N. Sen Sarma (Chem. Engng Dept., Jadavpur Univ., Calcutta, India). *Anal. Chem.*, 1960, **32** (6), 717–719.—The method depends on the titration with EDTA (disodium salt) of the blue soln. formed when acetone and an excess of NH_4SCN are added to a neutral or acetic acid soln. of Co^{II}. The end-point (colourless or pink) may be determined visually or photometrically. As little as 2·5 μg of Co per ml may be determined visually, or 1 μg per ml photometrically. Pyrophosphate, citrate, oxalate and cations other than alkali metals interfere; moderate amounts of orthophosphate, fluoride and tartrate can be tolerated. Modified procedures are described for the determination in the presence of alkaline-earth metals, Fe, Al, Cu and Ni. The best results are obtained when the soln. for titration contains 5% and 30 to 35%, respectively, of NH_4SCN and acetone, and <5 μg (visual) or 4 μg (photometric) of Co per ml.

R. E. E.

136. Determination of cadmium as impurity in nickel and cobalt by ion exchange. V. N. Pavlova and N. P. Strel'nikova. *Zavod. Lab.*, 1960, **26** (5), 536–537.—Cadmium is adsorbed on a column of ÉDÉ-10p from a soln. in 2 N HCl, whilst Ni, Co, Cu and Fe are not adsorbed; Cd is eluted with water and determined colorimetrically with dithizone.

G. S. SMITH

137. Determination of ruthenium in the presence of uranium by dithio-oxamide. V. I. Shlenskaya. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, II, No. 2, 69–72.—The method is based on the formation of a blue complex by ruthenium salts in aq. ethanolic HCl soln. with dithio-oxamide (**I**). If the ratio of Ru to U is $\simeq 1:100$, adopt the following procedure. Add conc. HCl (1 ml) and 0·1% ethanolic **I** soln. to the ruthenium salt soln. and heat it on a boiling-

water bath for 30 min. Make the soln. up to 25 ml with ethanol and measure the extinction at 610 to 660 mμ against an ethanolic **I** soln. (2 ml of 0·1% ethanolic **I** soln. per 25 ml of ethanol) as the blank. Beer's law is obeyed in the range 10 to 60 μg of Ru per 25 ml of soln. With $\lambda_{max.} = 650$ ($\epsilon_{max.} = 8500$) the sensitivity is 0·23 μg of Ru per ml. If the ratio of Ru to U is $>1:100$, adopt the following procedure. Add conc. HCl (1 ml) and 0·1% ethanolic **I** soln. (1 to 2 ml) to a soln. containing 10 to 50 μg of Ru. Heat the soln. for 5 min. on a boiling-water bath, allow it to cool and carry out a triple extraction with isoamyl alcohol $(5 + 5 + 5$ ml). Make the combined extracts up to 25 ml with isoamyl alcohol. Measure the extinction at 610 to 660 mμ against isoamyl alcohol as the blank soln. Beer's law is again obeyed for the same concn. range.

K. R. C.

138. Absorptiometric determination of palladium as its 2-mercapto-4,5-dimethylthiazole complex. A. J. Radford (Geological Survey Office, Dodoma, Tanganyika). *Analyst*, 1960, **85**, 445–448.—Ryan (*Ibid.*, 1951, **76**, 310; 1950, **75**, 557) states that the determination of Pd with 2-mercapto-4,5-dimethylthiazole is not affected by changes in acidity and concn. of NaCl. The method was studied in detail and the absorption measurements were extended into the u.v. region. When the complex is measured in the u.v. region with a Spekker absorptiometer and a mercury-vapour lamp there is an increase of 160% in sensitivity and the soln. obeys Beer's law. The acidity must be controlled when measurements are made in the u.v. region, but need not be when a tungsten-filament lamp is used. Addition of ethanol to prevent turbidity is not necessary. Nitric acid oxidises the reagent and must be removed.

A. O. JONES

139. New gravimetric methods for the micro-determination of osmium. P. Spacu and C. Gheorghiu (Inorg. Chem. Lab., Univ. "C.I. Parhon," Bucharest, Romania). *Z. anal. Chem.*, 1960, **174** (5), 340–345 (in German).—Three weighing forms for osmium are described—(a) $[OsCl_6]H_2.2NH_2C_6H_4-CH_2C_6H_5$ is pptd. from HCl soln. containing $[OsCl_6]^{2-}$ by acridine soln. (2% in acetic acid) and dried *in vacuo* after being washed with ethanol and ethyl ether; (b) $[OsBr_6]H_2.2NH_2C_6H_4CH_2C_6H_5$ is similarly pptd. from HBr soln. containing $[OsBr_6]^{2-}$, and can be dried at 110°; (c) $[Os(NH_2CSNH_2)_6][Cr(SCN)_6]$ is pptd. from H_2SO_4 or HCl soln. containing OsO_4 by acid thiourea soln. followed by $K_3[Cr(SCN)_6]$ soln. and can be dried at 105°. The preparation of $K_3[Cr(SCN)_6]$ is described. The pptn. of (a) and (b) can be carried out in the presence of the alkali metals, the alkaline-earth metals, Cu^{2+}, Fe^{3+}, Cr^{3+} and Co^{2+}; form (c) is used with soln. obtained by the distillation of OsO_4. All three pptn. are accurate to within $\simeq \pm 2\%$ for amounts of Os in the range 0·5 to 5 mg.

W. T. CARTER

140. Coulometric titration with electro-generated tin(II). Titration of platinum(IV). A. J. Bard (Univ. Texas, Austin, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 623–625.—Quadrivalent Pt is reduced only to Pt^{II} by an equivalent amount of Sn^{II} in an acid bromide medium. A slight excess of Sn^{II} is generated and back-titrated with electro-generated Br, with potentiometric or spectrophotometric end-point detection; 2 to 13 mg of Pt can be titrated with an average error of $\pm 0·5\%$ with

potentiometric end-point detection. Osmium, Ru, Cu, Fe and Au interfere and the Pt must then be separated; Pd^{II}, Rh^{III} and Ir^{IV} interfere only slightly. (*Cf.* Bard and Lingane, *Anal. Abstr.*, 1960, **7**, 511, 889.)　　　D. C. ARMSWORTH

141. Assay procedure for platinum in reforming catalysts. I. Maziekien, L. Ermanis and T. J. Walsh (Engelhard Industries, Inc., Newark, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 645–647.—The extinction of the Pt - SnCl$_2$ complex is measured. Fresh catalyst (3 g) is dissolved in HCl (1:1) (150 ml) by heating and the soln. is filtered into a 2-litre flask; AlCl$_3$ soln. (45·4% aq.) (100 ml), conc. HCl (650 ml) and SnCl$_2$ soln. [28% in HCl (1:1)] (60 ml) and water are added, the soln. is mixed and made up to vol. The extinction is read in a Corex cell in a recording spectrophotometer at 403 mμ, scanning from 375 to 425 mμ. Standard Pt soln. are measured alternately with sample soln. to provide the conversion factor. Spent catalyst is ignited to remove C and S, then dissolved in aqua regia, and the Pt is separated with H$_2$S or by reduction to metal with granulated zinc. The ppt. is dissolved in aqua regia, the HNO$_3$ is removed by evaporation, and the soln. is treated as described above. An alternative separation with zinc for use when large amounts of Fe or Mo are present, is also described. Full experimental details are given.　　R. E. E.

142. The heterometric determination of platinum, palladium and gold by titration with 1,10-phenanthroline. M. Bobtelsky and M. M. Cohen (Dept. of Inorg. and Anal. Chem., Hebrew Univ., Jerusalem). *Anal. Chim. Acta*, 1960, **22** (6), 532–538 (in English). —Instructions with experimental details are given for the determination of Pt^{IV}, Pd^{II} and Au^{III} as chlorides in thiocyanate or chloride soln. with 1,10-phenanthroline at pH 1 to 7. The procedures for the analysis of binary mixtures of these ions and also for the determination of 1,10-phenanthroline with PdCl$_2$ are described. The error, in most cases, is zero.　　　R. M. S. HALL

143. Measurement of thin metal layers. Fluorescent X-ray production by radio-isotope sources. G. B. Cook, C. E. Mellish and J. A. Payne (Isotope Res. Div., A.E.R.E., Harwell, England). *Anal. Chem.*, 1960, **32** (6), 590–593.—By choosing the correct method (either *K* X-ray excitation, *L* X-ray excitation or base-metal *K* X-ray absorption) and the correct source, it is usually possible to make the measurements sensitive at the thicknesses that are of interest. This flexibility is limited, however, by the absorptive properties of the metal under investigation. Measurements on chromium, copper and tin plate are reported and discussed.
　　　K. A. PROCTOR

144. Rapid automatic X-ray fluorescence analysis of mineral powders and solutions from the hydrometallurgical works of the Union Minière du Haut-Katanga. A. Hans (C.N.R.M., Section de Liège). *Chim. Anal.*, 1960, **42** (5), 236–244; (6) 282–286.— Copper, zinc and cobalt have been determined in cuprozinc and cuprocobalt minerals and concentrates. A tungsten or gold anti-cathode is used with voltages from 25 to 30 kV. The K_α lines of the elements are used for the determinations. The sample (2 g) is dissolved in acid and used as a liquid soln., or the powdered sample (0·2 g) is mixed with BaO$_2$ (2 g) and fused with borax (10 g) and used as a "solid" soln. By such means,

absorption effects are eliminated, and an internal standard is unnecessary. The calibration curves for the soln. method show slight curvature but not those for the borax-bead method. The ranges of concn. covered are—Zn, 0·05 to 60%; Cu, 0·05 to 50%; and Co, 0·05 to 5%. The method, suitable for routine process control, for the determination of Co (1·5 to 3%) and Cu (0·5 to 5%) in copper electrolysis soln. containing 0·5 to 5% of free H$_2$SO$_4$, involves irradiation with X-rays (Co K_α and Cu K_α, respectively) and measurement of the resulting fluorescence with a scintillation counter. The concn. of Cu has much less influence on the fluorescence of Co than has Co on that of Cu. To minimise these effects the soln. are diluted (1:10) and the free acid concn. is kept constant. The Co and Cu concn. can be rapidly determined with high precision by successive approximations to calibration curves.　　　　　R. M. S. HALL
　　　　　　　　　　　　J. P. STERN

145. Spectrochemical rubidium - strontium method for geological age determination. H.-J. Eichhoff (Inst. for Inorg. and Nuclear Chem., Johannes Gutenberg Univ., Mainz, Germany). *Appl. Spectroscopy*, 1960, **14** (3), 74–76.—A flame method is described for the determination of Rb and Sr in lepidolites after preliminary ion-exchange separations and with In as an internal standard. The abundance of ^{87}Sr is determined by excitation of the mineral in a hollow-cathode source, and the examination of the hyper-fine structure of Sr 4078 A, obtained by means of a Fabry-Perot etalon. Results of geological age determinations made by this method are in good agreement with those obtained by other methods and with existing geological theories.　　　　P. T. BEALE

146. Gravimetric method for the determination of barium oxide in Portland cement. C. L. Ford (A.S.T.M., 1916 Race Street, Philadelphia, Pa., U.S.A.). *Bull. A.S.T.M.*, 1960, (247), 77–80.— By the procedure given, from 0·01 to 0·20% of BaO may be determined with a max. error of 0·015%. The sample (1 g) is dissolved in hot dil. HCl, the soln. is diluted to ≃250 ml (to prevent subsequent pptn. of CaSO$_4$), then boiled, and BaSO$_4$ is pptd. by the addition of H$_2$SO$_4$ (1:1). The mixture is kept at ≃100° for 12 to 16 hr., then filtered, and the ppt. is ignited at 800° to 900° for 15 min.; HF is then added to decompose the HCl-insol. matter and volatilise any SiO$_2$. The residue is fused with K$_2$S$_2$O$_7$, the melt is extracted with H$_4$O (the BaSO$_4$ remains insol.), H$_2$SO$_4$ (1:1) is again added and, after 16 to 18 hr., the hot soln. is filtered through Whatman No. 42 paper. The BaSO$_4$ is washed, dried and ignited to const. wt. at 800° to 900°. The accuracy of the method was verified by analyses of mixtures containing known amounts of Ba.　　　　　W. J. BAKER

147. Quantitative analysis of radionuclides in process and environmental samples. A. L. Boni (Savannah River Plant, E.I. du Pont de Nemours & Co., Inc., Aiken, S.C., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 599–604.—An ion-exchange procedure, combined in some cases with radiochemical pptn., has been developed for the separation and recovery of Ru, Zr, Nb, Np, Co, Fe, Zn, Sr, rare-earth elements, Cr and Cs from natural materials. Iodine is eliminated by volatilisation. The recovery of individual isotopes is >95%, with a standard

deviation of $\pm 4\%$ ($P = 0.9$). The decontamination factors are $> 10^4$ for all ion-exchange separations and $> 10^3$ for gravimetric separations.

K. A. Proctor

See also Abstracts—**153**, Determination of Hg. **302**, Separation of O, N, CO_2 and N_2O by molecular sieve. **309**, Heavy metals in sugar syrups. **337**, Cu and Fe in water. **341**, Fission products in water. **363**, Chromatography of oxidisable metals in an inert atmosphere.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives, etc.

148. Interaction of beta-particles with matter. Quantitative determination of hydrogen, carbon, nitrogen, oxygen and fluorine materials. P. R. Gray, D. H. Clarey and W. H. Beamer (Radiochem. Lab., The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 582–589.—The use of β-ray back-scattering and β-ray transmission for the analysis of liquids is accurate, rapid, simple and non-destructive. Binary compounds, such as hydrocarbons, can be completely analysed by either method, whereas ternary compounds require intensity measurements by both methods for a complete quant. analysis. Compounds and mixtures containing more than three elements can only be analysed if quant. data are available for all the elements in excess of three. The standard deviation in the experimentally determined weight fraction of H in hydrocarbons and ternary compounds is $\simeq 0.03\%$. The precision is approximately the same for both the back-scatter and transmission instruments.

K. A. Proctor

149. Spectrographic method of determining traces of [metallic] impurities in organic compounds. Sh. I. Pelzulaev, L. K. Popova and R. L. Slyusareva. *Zavod. Lab.*, 1960, **26** (5), 552–553.—The sample (5 g) is mixed with 315 mg of spectrographically pure $SrSO_4$ and evaporated to dryness, the residue is treated with 0.5 ml of conc. H_2SO_4, the soln. is evaporated to fuming, and the residue is ignited and powdered. With difficultly volatile compounds the sample is mixed with $SrSO_4$ and 2 ml of conc. H_2SO_4 and evaporated nearly to dryness, followed by treatment with H_2SO_4 and HNO_3, evaporation, etc. The spectrum is excited in a d.c. arc between carbon electrodes, and 22 elements are determined by means of standards on a base of $SrSO_4$.

G. S. Smith

150. Analysis of ternary solvent mixtures. F. Oehme (Inst. Entwicklung chem.-phys. Analysenmethoden, Weilheim/Obb). *ChemikerZtg*, 1960, **84** (9), 291–294.—Since the determination of n components requires the measurement of $(n - 1)$ parameters, the measurement of the dielectric constant must be supplemented for such cases. Other suggested measurements are those of refractive index and density and a further extension is the measurement of water content by the Karl Fischer method before the determination of a single physical constant. A system of triangular co-ordinates and the resolution of a mixture by progressive separation of the components with a molecular sieve are described.

E. G. Cummins

151. Apparatus for hydrogen determination in hydrocarbons using β-radiation. M. Přibyl (Military Acad., Brno, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (5), 240–243.—The procedure previously described (*Anal. Abstr.*, 1960, **7**, 3785) is modified. The apparatus (illustrated) comprises a radiation source (20 mC of ^{90}Sr), a measuring and a reference ionisation chamber, and a measuring and an auxiliary wedge filter. The difference in the ionisation currents is measured by means of a recording galvanometer. The determination takes 20 min., and has an accuracy of $\pm 0.03\%$. Small amounts of O, N and S do not interfere, but corrections must be applied for larger amounts. Calibration is carried out with pure hydrocarbons. The apparatus can be adapted for continuous measurement, and the method is suitable for analysis of fuels and lubricating oils.

J. Zýka

152. Determination of sulphur in organic compounds with metallic silver as absorbent. A critical examination. J. P. Dixon (Shell Research Ltd., Thornton Res. Centre, Chester, England). *Talanta*, 1960, **4** (4), 221–226 (in English).—The sample is burnt by the "empty tube" method (*cf.* Ingram, *Anal. Abstr.*, 1956, **3**, 1745), the sulphur oxides produced are absorbed on electrolytically prepared silver and the Ag^+ equivalent to the S are extracted with boiling water and determined by potentiometric titration with 0.1 N KI, with glass and silver electrodes. In order to obtain blanks low enough for the determination of micro amounts of S, it is necessary to use boiled de-ionised water for the extraction, to wash the silver with ethanol and ethyl ether between analyses and to blanket it with nitrogen during cooling. With a 25-mg sample, 10 to 15 μg of S can be determined with a reproducibility of $\simeq 3\%$, but Cl interferes seriously and must be absent.

W. T. Carter

153. Determination of mercury in organomercury compounds and certain inorganic compounds. F. Martin and A. Floret (Soc. des Usines Chim., Rhône-Poulenc, Vitry-sur-Seine). *Bull. Soc. Chim. France*, 1960, (4), 610–613.—Two procedures are described. In the first the compound is placed between layers of CaS and heated in a slow stream of air. The vapours are passed through successive layers of granular CaO and silvered pumice (prep. described) at 750° to 800° and the Hg is absorbed in a tube containing gold leaf. The method is suitable for 1 to 20 mg of Hg. In the second, more generally applicable, procedure, the sample is pyrolysed in a stream of H and burnt in an oxyhydrogen flame as previously described (*Anal. Abstr.*, 1960, **7**, 559), with the modification that a portion of the O feeding the flame is bubbled through satd. bromine water. The $HgBr_2$ so formed is condensed with the water and received in a beaker containing 10 ml of water. The excess of Br in the condensate is removed by adding hydrazine hydrochloride, in small portions, till the colour is discharged, and 0.1 g in excess. After stirring for 2 min., 0.1 M EDTA (disodium salt) (10 ml) is added, followed by a vol. of pyridine equal to the total vol. of the soln., and the Hg is determined by potentiometric titration with 0.01 M Na diethyldithiocarbamate. Results by the two methods range from 98 to 101% of the theoretical.

R. E. E.

154. Gasometric determinations of certain organic nitrogen functions, and, more generally, of compounds capable of the quantitative liberation of a volume of gas. L. Nicolas and J. Mansel (Compagnie Franç. des Matières Colorantes, Usine d'Oissel, France). *Chim. Anal.*, 1960, **42** (4), 171–180; (5), 226–235.—An apparatus is described which permits N to be determined in a wide range of organic materials. An extension is provided above the nitrometer, so that the N evolved is measured in a graduated tube over water. The CO_2 used to expel air from the apparatus is obtained by heating $NaHCO_3$. The sample, dissolved in cooled H_2O and, if necessary, HCl or acetone, is kept cold in an ice bath before addition to the reaction vessel which contains the boiling decomposition reagents. For the determination of N in diazo compounds (*e.g.*, aliphatic and aromatic diazo compounds, stabilised diazonium salts, diazotates and triazenes), the sample (0·2 to 1 g) in aq. soln. is added to 150 ml of H_2SO_4 (30%) containing 1 g of Cu_2Cl_2. Excellent agreement is obtained with theoretical results, except in those few cases when another gas is evolved besides N. When this gas is identified and dissolved in a suitable absorbent (*e.g.*, hydrocarbons in ethanol), then the correct result is given. Procedures are also described for the determination of N in diazosulphonates and phenylhydrazine derivatives. Compounds containing the $-NH_2$ group (*e.g.*, *p*-nitroaniline) are determined after diazotisation, aromatic compounds containing the $-OH$ and $-NH_2$ groups by coupling with a known excess of a diazonium salt and determination of the excess, and compounds containing carbonyl groups (*e.g.*, aldehydes and ketones) by conversion into phenylhydrazones with a known excess of a phenylhydrazine derivative, and determination of the excess. A literature survey is given, with 99 references.
J. H. WATON
R. E. E.

155. Dinitrophenylation method for the microdetermination of amide nitrogen [in wool]. J. H. Bradbury (Div. of Textile Ind., Wool Res. Lab., C.S.I.R.O., Geelong, Australia). *Anal. Chim. Acta*, 1960, **22** (5), 444–448.—The procedure described is based on the reaction of NH_3 (derived from the protein by HCl hydrolysis) with 1-fluoro-2,4-dinitrobenzene to give 2,4-dinitroaniline (I), which is determined spectrophotometrically at 345 mμ after separation by paper chromatography. The yield of I is constant at 80% under the given conditions. The minimum amount of NH_3 necessary is $\simeq 2~\mu g$. With 0·5-mg wool samples the mean value was 7.4 ± 0.4 moles per 10^4 g of material (at least 4 determinations). Cuticle-rich samples may differ significantly from whole-fibre samples.
R. M. S. HALL

156. Quantitative analysis of monochloride mixtures by means of infra-red absorption spectra. V. I. Kolbasov, S. B. Bardenshtein and R. V. Dzhagatspanyan. *Zavod. Lab.*, 1960, **26** (5), 587–590.—The mixture of chlorides obtained by the chlorination of propylene is analysed by means of the absorption bands at 1875 cm^{-1} (allyl chloride), 880 cm^{-1} (2-chloropropene), 1319 cm^{-1} (*cis*-1-chloropropene), 855 cm^{-1} (n-propyl chloride) and 1060 cm^{-1} (isopropyl chloride).
G. S. SMITH

157. Micro-determination of ethanol in aqueous media. L. Bessot and M. Scemama (Lab. Phys., Fac. de Pharm., Paris, France). *Compt. Rend.*, 1960, **250** (23), 3897–3899.—The method of Nicloux is modified to avoid the preliminary distillation and the final titration with $Cr_2O_7^{2-}$. *Procedure*—Treat the sample with water (1 ml) and H_2SO_4 (1 ml) and heat to 85° for 3 hr. in a closed system that contains in a separate vessel 5 ml of 0·1 N $K_2Cr_2O_7$ in H_2SO_4 (1:1). The ethanol diffuses quantitatively into and reacts with the $K_2Cr_2O_7$. Determine the amount consumed, absorptiometrically, at 580 mμ. The error for ethanol (1·5 to 3·2 mg) is $< \pm 3\%$.
J. P. STERN

158. Studies on fermentation products. I. Fusel oil analysis by gas chromatography. Akira Kamibayashi, Makoto Miki and Hideo Ono. *Rep. Ferment. Res. Inst., Chiba*, 1960, (18), 101–110 (in Japanese).—The samples (0·1 to 0·3 ml), containing n-butanol as internal standard, were analysed in a Perkin-Elmer Vapour Fractometer, model 154-C, with thermal conductivity detection and He as carrier gas. Three columns (4 mm × 2 metres) were used, containing the following stationary phases on a support of diatomaceous earth, *viz*, (A) tetraethylene glycol dimethyl ether, (B) polyoxyethylene glycol 1500 and (C) dibenzyl ether. The operating temp., inlet pressures and flow-rates were, respectively, (A) 74°, 30 p.s.i. and 112·4 ml per min., (B) 100°, 15 p.s.i. and 76·4 ml per min. and (C) 78°, 15 p.s.i. and 83·7 ml per min. Alcohols were identified in the peak fractions as 3,5-dinitrobenzoates (ethanol, n-propanol and isobutyl alcohol), as the 3-nitrophthalate (isoamyl alcohol) or by optical activity (2-methylbutan-1-ol); isoamyl alcohol and 2-methylbutan-1-ol could not be separated on either column. Regression equations of the form $Y = aX + b$ were calculated, where Y is the ratio of wt. of alcohol to wt. of n-butanol, X is the ratio of the corresponding peak areas, and a and b are constants. Retention volumes relative to n-butanol on the three columns are tabulated for water and 11 alcohols. [From English summary.]
R. E. E.

159. Analysis of 1,2-glycols and polyhydric compounds. III. Direct iodimetric determination of glucose via the aldehyde formed by periodate oxidation. L. Maros and E. Schulek. *Magyar Kém. Foly.*, 1960, **66** (4), 147–149.—This is a version (in Hungarian) of the article published (in German) in *Acta Chim. Acad. Sci. Hung.*, 1959, **21**, 91; *cf. Anal. Abstr.*, 1960, **7**, 3307.

160. Microbiological assay for the quantitative determination of glycerol. L. Eidus, B. B. Diena, A. C. Maniar and L. Greenberg (Biologics Control Lab., Dept. of Nat. Health and Welfare, Ottawa, Canada). *Canad. J. Microbiol.*, 1960, **6** (3), 283–287.—The cylinder-plate method described is based on the observed inhibitory effect of glycerol on *Staphylococcus aureus*. Soln. of glycerol in the range of 20 to 80 mg per ml are incubated for 16 to 18 hr. on a previously seeded medium (prep. described), and the amount corresponding to zone size is read from a standard curve. Variation of results from a known concn. was $\simeq 12\%$, as compared with $\simeq 10\%$ for a chemical method. Glucose, fructose, maltose, mannitol, mannose, sucrose and trehalose interfered, but nine other carbohydrates tested did not. The method is applicable to culture fluids after bacterial growth.
M. A. ELLIS

161. Microbiological assay of glucose as applied to starch hydrolysates. M. D. Smith, M. W. Radomski and J. J. Kagan (Univ. Toronto, Ontario, Canada). *Anal. Chem.*, 1960, **32** (6), 678–680.—The

method is based on the ability of *Lactobacillus casei* to ferment glucose selectively in a mixture of the sugars found in starch hydrolysates. The response is determined by titration with standard alkali soln. and the dose - response curve is linear up to 4 mg of glucose. A heavy-inoculum technique shortens the incubation time to 2 hr. and is sensitive to small quantities of glucose. Recoveries of glucose added to hydrolysates averaged 100·1% at the 1-mg level, and a mean difference of ±3·0% with a standard deviation of 0·148 was obtained from 50 sets of duplicate titrations.   G. P. Cook

162. Gas-chromatographic retention time of form-aldehyde. A. I. Schepartz and P. E. McDowell (Agric. Res. Service, U.S. Dept. of Agric., Phila-delphia, Pa.). *Anal. Chem.*, 1960, **32** (6), 723.—A retention time of 2·3 min. was obtained for form-aldehyde on a 10-ft. column of Carbowax 20M on Chromosorb (35 to 80 mesh) at 90°, with a helium flow-rate of 32 ml per min. In determining this retention time the flash-exchange technique of Ralls (*Anal. Abstr.*, 1960, **7**, 4825) was used.
                                            K. A. Proctor

163. Separation and identification of C8 aldehydes. Use of gas - liquid chromatography, nuclear magnetic resonance, and infra-red spectroscopy. J. S. Mat-thews, F. H. Burow and R. E. Snyder (Gulf Res. and Devel. Co., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 691–693.—In the method described, individual isomers or groups of isomers are separated by preparative gas chromatography. Nuclear magnetic resonance is then used to deter-mine the number of CH_3, CH_2 and CH groups, and i.r. spectroscopy to indicate the presence of $(CH_3)_3$ groups ($n \ll 3$). The aldehyde is finally reduced to a hydrocarbon, which is identified by comparison with suitable standards by gas - liquid chromato-graphy.   K. A. Proctor

164. Chromatographic separation of 2,4-dinitro-phenylhydrazine derivatives of highly oxygenated carbonyl compounds. M. L. Wolfrom and G. P. Arsenault (Ohio State Univ., Columbus, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 693–695.—The chromato-graphic separation of the 2,4-dinitrophenylhydrazine derivatives of highly oxygenated 2- and 3-carbon carbonyl compounds was effected on a column of silicic acid - Celite (5:1, containing 8% of H_2O).
                                            G. P. Cook

165. Paper chromatography and structural rela-tionships of organic acids. J. R. Howe (Low Temp. Res. Sta., Cambridge, England). *J. Chromato-graphy*, 1960, **3** (5), 389–405 (in English).—The R_F and R_M [= $\log(1/R_F - 1)$] values for 111 organic acids were determined in an alkaline and an acid solvent, *viz*, n-propanol - 2 N aq. NH_3 (7:3) and n-propanol - aq. satd. soln. of SO_2 (7:3). Results are discussed in relation to the structures of the acids, which included several homologous series. A plot of R_F values in acid solvent against those in alkaline solvent shows that distinct groupings are obtained according to the number of carboxyl groups in the molecule, whereas neutral compounds have similar R_F values in both solvents. The R_M values for several homologous series when plotted against number of carbon atoms give approx. linear relationships up to 8 carbon atoms in both solvents. Definite groupings also occur if the difference between the R_M value in alkaline solvent and that in acid solvent is plotted against the R_M values in alkaline solvent, and homologous series form sub-groups. A study of the R_M value could

therefore be used to give an indication of the presence of substituent groups such as $-CO_2H$, $-OH$, alkyl, $-NH_2$ and bromo– in an unknown acid and the manner in which they are arranged.
                                            S. M. Marsh

166. Gas-chromatographic separation of volatile organic acids in presence of water. I. R. Hunter, V. H. Ortegren and J. W. Pence (U.S. Dept. Agric., Albany, Calif., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 682–684.—Volatile organic acids (C_2 to C_6) are separated at 125° by using an adipate polyester of diethylene glycol (LAC-1-R 296) as the liquid phase and He as the carrier gas. The fractions emerging from the column are oxidised to CO_2 which is then measured in a thermal conductivity cell; H_2O does not affect the separation, and is removed by a drying tower placed before the conductivity cell.
                                            G. P. Cook

167. Determination of oxalates in the presence of chromium. V. F. Saksin (Technol. Inst. Yaroslav). *Zavod. Lab.*, 1960, **26** (5), 546–547.—An accurately measured vol. of 0·1 N $KMnO_4$ is treated with excess of $MnSO_4$ followed, after 5 min., by 15 to 20 ml of 2 N H_2SO_4 to give H_2MnO_3, which will oxidise oxalate but not Cr^{3+}. The soln. is boiled for 20 to 25 min. with a known vol. of the soln. to be analysed, then cooled, mixed with sufficient 20% KI soln. to react with the excess of H_2MnO_3, and titrated with 0·1 N $Na_2S_2O_3$.   G. S. Smith

168. Differential thermal analysis and thermo-gravimetry of some salts of guanidine and related compounds. M. I. Fauth (U.S. Naval Propellent Plant, Indian Head, Md.). *Anal. Chem.*, 1960, **32** (6), 655–657.—The picrates and styphnates of hydrazine, guanidine, aminoguanidine, guanylurea, *N*-methylguanidine and *N*-ethylguanidine were prepared and their impact sensitivities and thermal behaviour under rapid heating studied. The temp. range covered was from room temp. to the point of decomposition of the compound being investigated. Differential thermal analysis curves and thermo-gravimetric results are given for 16 compounds. At rapid heating rates (8° per min.) considerably lower decomposition temp. may be expected than would be indicated by m.p. and other values obtained by slow heating.   G. P. Cook

169. Colour reactions for organophosphorus com-pounds. H. Hecker and F. Hein (Inst. f. Anorg. Chem., Friedrich Schiller Univ., Jena). *Z. anal. Chem.*, 1960, **174** (5), 354–359 (in German).—(*a*)—Phosphine oxides and dialkylphosphinic acids give deep-blue colorations when heated with an-hydrous $NiBr_2$ in toluene. Some phosphine sul-phides and tertiary phosphines give green colours. Acetone and ethyl methyl ketone interfere. (*b*)—Alkylphosphine oxides and tetra-alkylphosphonium salts give a deep-violet colour with *m*-dinitrobenzene and 35% NaOH soln. (*c*)—Tertiary phosphines give a red - violet colour with 3,5-dinitrobenzoic acid, triethylphosphine oxide and CCl_4. (*d*)—Ali-phatic phosphine sulphides give a green colour changing to blue when heated on a water bath with vanillin and conc. HCl. (*e*)—Aliphatic phosphine oxides give a brown or green coloration with CS_2, NaOH and a molybdate.   W. T. Carter

170. Detection of phosphate esters on paper chromatograms. F. E. G. Harrap (Levington Res. Sta., Ipswich, Suffolk, England). *Analyst*, 1960, **85**, 452.—Phosphate esters, *e.g.*, fructose 1,6-diphosphate, glucose 1-phosphate, glucose 6-phos-

phate and phytic acid are usually detected on filter-paper by hydrolysis to H_3PO_4, which is treated with an acid molybdate spray or dip; the complex formed is then reduced to a blue compound when the paper is treated with H_2S or exposed to u.v. light. After either treatment the paper gradually becomes blue, and later identification of faintly coloured spots becomes difficult. Also certain naturally occurring acids (*e.g.*, citric, tartaric and ascorbic acids) form a blue colour slowly. To overcome these disadvantages the filter-paper is dried in air and dipped in a mixture of 5 ml each of 60% $HClO_4$ and 20% molybdate soln., 10 ml of N HCl and 80 ml of acetone. The paper, dried in air, is exposed to u.v. radiation then immediately dipped in a 2·5% (w/v) soln. of benzoin α-oxime in methanol and dried. This provides the paper with a background of a white ppt. stable for some weeks. The acids (*supra*) in 1% soln. do not form visible spots on this background.   A. O. JONES

171. Rapid method for qualitative analysis of volatile mercaptan [thiol] mixtures. J. F. Carson, W. J. Weston and J. W. Ralls (Western Utilisation Res. & Devel. Div., U.S. Dept. Agric., Albany, Calif.). *Nature*, 1960, **186**, 801.—Equal weights (3 to 5 mg) of a mixture of mercuric mercaptides and warm toluene-3,4-dithiol are centrifuged to produce a slurry in a capillary tube. The tube is connected to a gas-chromatography unit and the lower end of the tube is heated at 245° to 260° for 15 sec. when an exchange reaction occurs and C_1 to C_4 saturated thiols flash into the unit for separation and identification. Only highly volatile thiols give good detector response.   A. C. R. HARTLEY

172. A new volumetric method for the estimation of thiourea. Prakash Chandra Gupta (Coll. of Sci., Hindu Univ., Varanasi). *J. Indian Chem. Soc.*, 1960, **37** (4), 213–216.—Thiourea is quant. oxidised to urea and sulphate by iodine in the presence of $NaHCO_3$. Standard iodine soln. of suitable concn. (20 ml) is mixed with 10% $NaHCO_3$ soln. (20 ml) and titrated with thiourea soln. The end-point may be detected visually (starch indicator) or potentiometrically. Alternatively, the thiourea soln. may be mixed with $NaHCO_3$ soln. and a known excess of iodine soln. and the excess back-titrated potentiometrically with standard arsenous oxide soln.   R. E. E.

173. Analysis of dithiocarbamates. G. Balif (Inst. Agron. Res., Acad. R.P.R.). *Rev. Chim., Bucharest*, 1960, **11** (6), 338–341.—A review of current methods is given, with 18 references.
                                         H. SHER

174. Determination of water-extractable sodium in liquid hydrocarbons. K. H. Nelson and M. D. Grimes (Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 594–595.—The Na is extracted by shaking the hydrocarbon with an equal volume of water and is determined in the aq. phase by flame photometry. The relative error is ±4% and the precision is ±2%, when applied to samples boiling in the range 135° to 900° F.   G. P. COOK

175. Colorimetric determination of benzene and chlorobenzene when present together. M. Mirjolet and C. Domange (Univ. Nancy, France). *Bull. Soc. Pharm. Nancy*, 1959, (41), 23–29.—Mixtures of benzene and chlorobenzene (I) are sometimes used as solvents for gum rubber in rubber cements. Nitration yields *m*-dinitro-derivatives in both cases,

and when the product is dissolved in ethyl methyl ketone and shaken with conc. aq. KOH soln. a violet colour appears. The max. absorption produced by benzene is at 570 mμ, while the max. of the weaker absorption produced by I is at 540 mμ. Attempts to distinguish colorimetrically between benzene and I have not given satisfactory results. However, it was found that, by boiling with Na_2CO_3, the 1-chloro-2,4-dinitrobenzene produced by nitration of I was completely converted into 2,4-dinitro-phenol, which gave max. absorption at 420 mμ, permitting sharp differentiation from benzene, since 2,4-dinitrobenzene was not affected by the Na_2CO_3. For small amounts of benzene and I (singly or together) the extinctions of the coloured soln. followed the Beer - Lambert law.
                                      CHEM. ABSTR.

176. Determination of polycyclic aromatic hydrocarbons in tobacco smoke. A. Candeli, A. J. Lindsey and K. Persaud (Dept. of Chem., Sir John Cass College, London). *Anal. Chim. Acta*, 1960, **22** (5), 458–461.—Previously described apparatus and methods (chromatographic and u.v. spectrophotometric) (*cf.* Lindsey, *Anal. Chim. Acta*, 1959, **20**, 175; **21**, 101; and Commins *et al.*, *Brit. J. Cancer*, 1955, **9**, 304) have been improved.
                                      R. M. S. HALL

177. Relative detector response in gas chromatography. II. Benzene hydrocarbons, phenols and phenol ethers. G. R. Jamieson (Chem. Dept., Paisley Tech. Coll., Scotland). *J. Chromatography*, 1960, **3** (5), 494–496 (in English).—Determination of the relative detector response of homologous series of benzene hydrocarbons, phenols and phenol ethers shows that there is an increase in response with an increase in molecular weight and for isomers there is a decrease in response with increase in chain-branching. Lower relative detector responses are given by *ortho*-isomers than by either *meta*- or *para*-isomers. If a compound has a group that forms part of a side-chain its response is similar to that of the isomer that has the group attached to the benzene ring. The introduction of a hydroxyl or carboxyl group lowers the relative detector response, but the introduction of a chloro-group into a phenol or phenol ether has little effect.   S. M. MARSH

178. Neutral silver nitrate as a reagent in the chromatographic characterisation of phenolic compounds. W. J. Burke, A. D. Potter and R. M. Parkhurst (Univ. Utah, Salt Lake City, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 727–728.—A neutral soln. of $AgNO_3$ in aq. acetone gives characteristic colour reactions with various phenols and certain of their ether derivatives. The time required to develop the colour is also a characteristic of the compound involved. R_F values in 5 solvent systems, colours produced, and colour-development times for 37 substances are listed.   G. P. COOK

179. Infra-red spectra of 1,4-benzoquinones. R. L. Edwards, J. Keighley and D. G. Lewis (Inst. Technol., Bradford, England). *J. Appl. Chem.*, 1960, **10** (6), 246–249.—Data are given for twenty 1,4-benzoquinones. Some derivatives of these compounds occur as pigments in fungi.

180. Simple specific test for inner-ring o-quinones. E. Sawicki and W. Elbert (Air Pollution Engng Res., Robert A. Taft San. Engng Centre, Cincinnati, Ohio, U.S.A.). *Anal. Chim. Acta*, 1960, **22** (5), 448–451.—The quinone is treated with 3,4-dimethoxy-aniline in hot acetic acid to give blue to green

compounds absorbing at 590 to 615 mμ. A spot-test procedure is described. Benzil, chloranil, fluorenone, acenaphthenequinone, anthraquinone and benzanthrone give negative results; 1,4-naphthaquinones give a scarlet coloration. The identification limits are 5 to 20 μg. The results of colour tests with o-quinones in H_2SO_4 are also reported.   R. M. S. HALL

181. **Determination of aniline in N-ethylaniline.** K. B. Whetsel, W. E. Roberson and M. W. Krell (Tennessee Eastman Co., Kingsport, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 730.—Infra-red data are given (*cf. Anal. Abstr.*, 1959, **6**, 1380).

182. **Analysis of mixtures of aniline and N-ethyl-aniline.** K. B. Whetsel, W. E. Roberson and M. W. Krell (Tennessee Eastman Co., Kingsport, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 730.—Infra-red data are given (*cf. Anal. Abstr.*, 1959, **6**, 1380).

183. **Analysis of mixtures of N-ethyl-m-toluidine and N-(2,3-dihydroxypropyl)-N-ethyl-m-toluidine.** K. B. Whetsel, W. E. Roberson and M. W. Krell (Tennessee Eastman Co., Kingsport, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 730.—Infra-red data are given (*cf. Anal. Abstr.*, 1957, **4**, 4028).

184. **Photometric titration of weak bases in non-aqueous media.** L. E. I. Hummelstedt and D. N. Hume (M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 576–581.—Weak organic bases are determined by photometric titration with $HClO_4$ in acetic acid as the titrant and acetic acid or methyl cyanide as the solvent. Differential titration of 4 components in a mixture is possible. Titration is normally carried out at the 10^{-8} M level but many bases can be successfully measured at the 10^{-4} M level. Wavelength data are given for 18 aromatic compounds. The deviation is generally $< \pm 5\%$.   G. P. COOK

185. **Studies on the analysis of drugs by the use of infra-red spectra. II. Assay of naphthols.** Michio Suzuki, Eizo Nakamura and Yuzo Nagase (Coll. of Pharm., Kashiwagi, Shinjuku-ku, Tokyo). *J. Pharm. Soc. Japan*, 1959, **79** (9), 1145–1148.—The determination of 1- (I) and 2-naphthol (II) in a binary mixture was attempted by infra-red spectra. The determination was made in CS_2 and in KBr discs by means of the bands at 770, 790 and 1083 cm⁻¹ for **I** in CS_2, 765 and 790 for **I** in KBr, 744 and 837 for **II** in CS_2, and 815 and 843 for **II** in KBr. The determination of **I** in **II** was unsuccessful in CS_2, owing to the slight solubility of **II**. The absorption of **II** in the soln. at 837 mμ is variable owing to its intermolecular action with **I**. The standard deviations were $< 1 \cdot 18\%$ and no significant difference was found between results obtained with CS_2 soln. and KBr discs.   S. NATORI

186. **Fluorimetry. III. Fluorimetric determination of naphthionic acid by means of short-wave ultra-violet light (313 mμ).** J. Eisenbrand and H. Meyer (Chem. Untersuchungsamt, Saarbrücken, Saarland). *Z. anal. Chem.*, 1960, **174** (6), 414–418.—The absorption spectrum of naphthionic acid [1-naphthylamine-4-sulphonic acid] (**I**) has a max. between 315 and 325 mμ and the fluorescence spectrum has a max. at 425 mμ. The Hg line at 313 mμ is useful for fluorescence excitation. Full fluorescence is attained between pH 6 and 8, and the log fluorescence vs. log concn. curve is linear in the concn. range $1 \cdot 6 \times 10^{-9}$ to 1×10^{-6} g per ml.

The method is used for determining **I** in the presence of 2-amino-1-naphthol-4-sulphonic acid in the enzymic degradation of Azorubine S (C.I. Food Red 3). The sensitivity is 0·001 μg per ml.   J. P. STERN

187. **The separation of some terpenoid compounds by gas - liquid chromatography.** E. von Rudloff. *Canad. J. Chem.*, 1960, **38** (5), 631–640.—The gas - liquid chromatographic separation of mixtures of some terpene hydrocarbons, their oxygenated derivatives, two sesquiterpene alcohols and three monophenols on a variety of columns was studied. The best solid support was Chromosorb W, a flux-calcined diatomaceous earth, which enabled separation to be made at high temp. without causing decomposition of the compounds. The effect of temp. and sample size on the degree of separation was considerable; low temp. gave the best results and sample sizes above 3 μl caused delays in the retention times and broadened the peaks. Monoterpene hydrocarbons were best separated on columns with squalene, Carbowax 400, Craig polyesters, and polyphenyl ether as the liquid phases at temp. below 130°. At higher temp. the polyester and polyphenyl ether columns were most suitable for the separation of oxygenated terpenes, sesquiterpene alcohols and monophenols. The adipic acid-polyoxyethylene glycol polyester column was the most suitable for all the compounds tested.   G. P. COOK

188. **Modified Ehrlich's reagent for measurement of indolic compounds.** M. Knowlton, F. C. Dohan and H. Sprince (Veterans Admin. Hosp., Coatesville, Pa., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 666–668.—The sensitivity and stability of the Ehrlich reaction are improved by the use of a nearly saturated soln. of p-dimethylaminobenzaldehyde in 12 N HCl as the reagent. Absorbance data are given for several indolic compounds and the concn. curves follow Beer's law, except that for indole, which deviates slightly. Concn. as low as $2 \cdot 5 \times 10^{-6}$ mole can be determined and the coeff. of variation range from 0·29 to 5·68.   G. P. COOK

189. **Determination of phenothiazine in commercial preparations by chromatography.** D. Gunew (Central Res. Lab., Imperial Chemical Industries of Australia and New Zealand Ltd., Newson St., Ascot Vale, Melbourne, Australia). *Analyst*, 1960, **85**, 360–364.—Sufficient sample to produce 25 to 35 mg of phenothiazine per ml of final soln. is extracted in a modified Soxhlet apparatus (described) with dichloromethane. The column is a mixture of silica gel (15 g), Magnesol (15 g), copper powder (1 g, prep. described) and the solvent mixture [dichloromethane - n-hexane (1:9)] (120 ml) in a water-jacketed chromatographic tube maintained at 14° to 16° surmounted by a reservoir of the solvent mixture. The sample soln. (2 ml) is applied to the column by means of a delivery tube, and the column is eluted under pressure of nitrogen with 800 ml of the solvent, and 10-ml fractions are collected. The solvent is evaporated from each fraction individually under reduced pressure at 30° to 35°. The phenothiazine-containing fractions (recognisable visually) are rinsed with dichloromethane into a tared flask, the solvent is removed and the residue is weighed and its m.p. determined. Since Magnesol is not now obtainable, Florisil (200 to 300 mesh) can be used in its place.   A. O. JONES

190. New photometric method for the determination of diphenylamine in phenothiazine. V. Pelloni and M. Sterescu. *Rev. Chim., Bucharest,* 1960, **11** (6), 346.—The proposed method is based on the colour reaction of diphenylamine (**I**) with *p*-dimethylaminobenzaldehyde (**II**) in the presence of oxidising agents. *Procedure*—A standard curve is prepared by dissolving 0·09 g of pure phenothiazine and 0·01 g of **I** in 100 ml of methanol - water (1:4). The soln. is centrifuged and filtered, and aliquots corresponding to 25 to 100 μg of **I** are taken. To each is added 2 ml of 10 N H_2SO_4, 4 ml of reagent (38 ml of conc. H_2SO_4, 37 ml of water and 0·125 g of **II**) and 6 drops of H_2O_2 soln. (5 ml of water and 2 drops of H_2O_2). The extinction is read after 20 min. at 570 mμ in a 1-cm cell against water. For the determination, 0·1 g of crude phenothiazine is dissolved in 100 ml of aq. methanol and treated as described above, and the extinction is compared with the standard curve. The error is ±5%.

H. SHER

191. Determination of 1,2-diphenylpyrazolidine-3,5-dione and its derivatives. M. Konupčík, F. Kupčík and M. Liška (Dept. Tech. Control, Farmakon, Olomouc, Czechoslovakia). *Českosl. Farm.,* 1960, **9** (5), 236–238.—Two acidimetric methods and one bromimetric method are described for the titration of 1,2-diphenylpyrazolidine-3,5-dione and 4-(3-oxobutyl)-1,2-diphenylpyrazolidine-3,5-dione (**I**) in non-aqueous solvents. *Acidimetric procedures*—(*i*) The sample (0·2 to 0·3 g) is dissolved in $CHCl_3$ or dioxan (20 ml, neutralised to phenol-phthalein) and titrated with 0·05 N Na methoxide, with phenolphthalein as indicator. (*ii*) The sample (0·4 to 0·5 g) is dissolved in dioxan (25 ml) and titrated with 0·1 N NaOH. The errors are ±0·15% and 0·05%, respectively. *Bromimetric procedure*—The sample (0·2 to 0·4 g) is dissolved in glacial acetic acid, conc. HCl (5 ml) and 20% KBr soln. (5 ml) are added, and the mixture is titrated potentiometrically with 0·1 N $KBrO_3$ (*cf.* Jančík *et al., Anal. Abstr.,* 1958, **5**, 246). The error is 0·1%. Only the bromimetric procedure is satisfactory for the determination of the sodium salt of **I**.

J. ZÝKA

192. Separation of 1,2-diphenylpyrazolidine-3,5-dione derivatives by paper chromatography. M. Konupčík, F. Kupčík and M. Liška (Dept. Tech. Control, Farmakon, Olomouc, Czechoslovakia). *Českosl. Farm.,* 1960, **9** (5), 239–241.—*Procedure*—Place the soln. of the sample (50 to 100 μg) in $CHCl_3$ on Whatman No. 1 paper and then impregnate the paper with formamide - ethanol (1:1) containing 5% of ammonium formate. After 10 min. separate chromatographically with benzene - cyclohexane (4:1 or 3:2); after 3 hr. dry *in vacuo* at 50° and detect by spraying with a freshly prepared mixture of 15% $FeCl_3$ soln. and 1% $K_3Fe(CN)_6$ soln. (1:1). R_F values are tabulated.

J. ZÝKA

193. New method for the determination of pyrazol-anthrone (6-oxoanthra[1,9-cd]pyrazole). M. Matrka and F. Navrátil (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl,* 1960, **10** (5), 248–249.—*Procedure*—Dissolve 6-oxoanthra-[1,9-cd]pyrazole (0·05 g) in ethanol (10 ml) and N NaOH (10 ml) and dilute with H_2O to 100 ml. Dilute 1 ml of this soln. to 10 ml with Britton - Robinson buffer soln. (pH 11·98), remove O with a stream of N and register the polarographic wave ($E_{\frac{1}{2}} = -1·12$ V). Compare with a calibration curve.

J. ZÝKA

194. Molecular sieve adsorption. Application to hydrocarbon-type analysis. J. G. O'Connor and M. S. Norris (Gulf Res. and Dev. Co., Pittsburgh, Pa., U.S.A.). *Anal. Chem.,* 1960, **32** (6), 701–706.—Normal hydrocarbons are determined in petroleum distillates boiling between 100° and 650° F by means of powdered molecular sieves, type 5-A, without previous fractionation of the distillates into narrow boiling-ranges. The sample is weighed into a column containing the sieves and the non-adsorbed hydrocarbons are eluted with isopentane. The adsorbed hydrocarbons are determined by weighing the column after removal of the excess of eluent, and can be removed from the column by extraction into n-pentane. Various fractions are analysed by mass spectrometry. A schematic diagram illustrates the various steps in the analysis of samples in conjunction with a silica gel separation technique. The accuracy, as standard deviation, is within ±0·8% of the normal hydrocarbon content of the sample.

G. P. COOK

195. Polarographic determination of manganese in gasoline. Triethanolamine complexes of manganese(II and III) and lead(II). E. R. Nightingale, jun., G. W. Wilcox and A. D. Zielinski (Univ. of Nebraska, Lincoln, U.S.A.). *Anal. Chem.,* 1960, **32** (6), 625–628.—The organomanganese compounds are decomposed by exposing the sample to u.v. light while it is being heated under reflux with conc. HCl. The Mn in the aq. extract is then determined polarographically as the triethanolamine complex at $-0·7$ V *vs.* the S.C.E.; Pb is determined at $-0·6$ V *vs.* the S.C.E. on another portion of the extract. When applied to petroleum-base stocks the standard deviation from the amount present was 5·2% for Mn and 1·6% for Pb. For pure iso-octane or aq. standards the average deviation was < ±0·5%.

G. P. COOK

196. Determination of polynuclear aromatic hydrocarbons in petroleum waxes. L. B. Nelson and D. J. Stormont (Socony Mobil Oil Co., Brooklyn, N.Y., U.S.A.). *Chem. & Ind.,* 1960, (20), 561.—A procedure is given for the separation and determination of tetracyclic and higher aromatic hydrocarbons in concn. of 10^{-3} to 10^{-9} in petroleum wax, with recoveries of 45 to 65%. The molten wax (100 g) is passed through a heated column (15 cm × 4·5 cm) of activated Al_2O_3 or silica gel which retains the polycyclic aromatics. After elution of the residual wax with iso-octane and extraction (in a Soxhlet thimble) of the extruded adsorbent with benzene - ethanol under N, the polycyclic aromatics can be further concentrated by making five partition extractions of the residue (dissolved in 50 to 100 ml of cyclohexane) in the thimble with nitromethane. The residue from the partition is then submitted to one-dimensional ascending paper chromatography with methanol - ethyl ether - water (4:4:1) as solvent on S. & S. 2043b acetylated paper. Sections of the chromatogram are cut out, eluted with cyclohexane - ethanol (1:1) and the u.v. spectra of the extracts are recorded. When necessary, fractions are re-examined by chromatography on Whatman No. 1 paper, with an iso-octane - dimethylformamide system, followed by comparison of R_F values (referred to benzo[a]pyrene, fluorescence colour and u.v. spectra.

W. J. BAKER

197. Separation of polycyclic aromatic hydrocarbons in complex mixtures. Chromatographic determination of trace amounts in petroleum waxes. W. Lijinsky (Chicago Med. Sch., Ill., U.S.A.).

Anal. Chem., 1960, **32** (6), 684–687.—Four poly-cyclic aromatic hydrocarbons, *viz*, benzo[*a*]pyrene, dibenz[*a*,*h*]anthracene, benz[*a*]anthracene and chrysene, added to petroleum waxes both individually and combined, were recovered by chromatography on magnesium oxide mixed with Celite, followed by paper chromatography with different solvent systems. Recovery ranged from 35% for benzo-[*a*]pyrene to 94% for dibenz[*a*,*h*]anthracene in the concn. range 0·01 to 1 p.p.m. G. P. COOK

198. Direct determination of traces of total oxygen in naphthas. I. J. Oita (Res. and Develop. Dept., Standard Oil Co., Whiting, Ind., U.S.A.). *Anal. Chim. Acta*, 1960, **22** (5), 439–443.—Further amendments to the modified Schütze method (*cf.* Oita and Conway, *Anal. Chem.*, 1954, **26**, 600) have been made. These include the handling of the sample in a quartz hollow spiral, controlled magnetically. Full details of the modified pyrolysis tube and operating instructions are given. For a synthetic mixture (cetyl alcohol in iso-octane) the value found was 157 ± 14 p.p.m. (3 determinations), calculated 144. For routine samples the deviations from the mean (duplicates) were about ±20%. R. M. S. HALL

199. Identification of Teepol and sodium lauryl sulphate in a commercial detergent mixture. B. Sewell. *Lab. Practice*, 1960, **9** (6), 381.—About 1 g of mixed alcohols, obtained by hydrolysing the detergent mixture by heating it under reflux with 3 N HCl for 30 min., is dissolved in 15 ml of dry benzene and heated under reflux for 2 hr. with 1·5 g of 3,5-dinitrobenzoyl chloride and 5 ml of pyridine. The mixture is dissolved in ethyl ether (50 ml) and extracted several times with 6 N HCl to remove pyridine and then with 4 N NaOH to remove 3,5-dinitrobenzoic acid. The ether soln. is washed free from alkali with water, dried over Na$_2$SO$_4$, and evaporated to dryness. The ester is then crystallised from light petroleum - ethanol (3:4). About 0·05 g of the crystals is dissolved in 5 ml of benzene - acetone (1:1) and paper chromatography is carried out with benzene as solvent until the front has ascended by 15 to 20 cm. After drying, the paper is sprayed with satd. ethanolic hydroxyammonium chloride soln. and dried. A cold conc. ethanolic NaOH soln. is then sprayed on. A yellow band near the solvent front indicates Na lauryl sulphate in the original sample; a sharp yellow band near the base-line indicates Teepol in the original product. The presence of an alkylaryl-sulphonate does not cause interference, since it is not hydrolysed under these conditions. N. E.

200. Methods of detection of the adulteration of volatile oils with ethanol. B. Kamiński and O. Dytkowska (Drug Res. Inst., Warsaw). *Acta Polon. Pharm.*, 1960, **17** (3), 213–219.—The methods of Pharm. Polon. III for the detection of ethanol in volatile oils are critically studied. A review of other published methods is given. The best test for the detection of a small amount of alcohol in essential oils is that of Pharm. Ned. VI, *viz*, the determination of refractive indices before and after removal of ethanol from the sample by shaking with satd. aq. NaCl soln. and drying with anhyd. Na$_2$SO$_4$. If the values differ by >0·001 the presence of ethanol is indicated. (19 references.) B. K.

201. Analysis and composition of oil of lemon by gas - liquid chromatography. R. A. Bernhard (Dept. of Food Sci. and Technol., Univ. of Calif.,

Davis, U.S.A.). *J. Chromatography*, 1960, **3** (5), 471–476 (in English).—Cold-pressed Californian lemon oil has been analysed by gas - liquid chromatography with LAC-2-R446 (adipate polyester of diethylene glycol partially cross-linked with penta-erythritol) and LAC-4-R777 (succinate polyester of diethylene glycol) as stationary liquid phases. Compounds identified by the corrected retention volumes method were—α-pinene, β-pinene, myrcene, (+)-limonene, n-heptanal, 1,8-cineole, γ-terpinene, n-hexanol, n-octanol, methylheptenone, n-nonanal, linalol, n-decanal, citronellyl acetate, citronellal, n-undecanal, tetrahydrogeraniol, decyl acetate, geraniol, α-terpineol, citronellol, neral, geranyl acetate, citral and (+)-carvone. S. M. MARSH

202. Determination of carbonyl groups in cellulose. H. Stübchen-Kirchner (Inst. f. Chemie u. chem. Technol. des Papieres u. des Zellstoffes, Tech. Hochschule, Graz). *Öst. ChemZtg*, 1960, **61** (5), 132–141.—A comprehensive review, with 60 references, is presented. S. M. MARSH

203. Rapid method for the determination of the acetic acid content of cellulose acetates. K. H. Bischoff and K. J. Linow (Dtsch. Akad. Wiss., Berlin). *Faserforsch. u. Textiltech.*, 1960, **11** (5), 245.—In Batt and Penche's method (*Bull. Soc. Chim. France*, 1929, **45**, 152) the cellulose ester is dissolved in pyridine, but cellulose acetates with high contents (59 to 62·5%) of acetate are not completely soluble. This can be overcome by shaking the cellulose acetate (0·4 g) with dichloromethane (20 ml) for 30 min. then adding pyridine (40 ml) and 0·5 N NaOH (30 ml). N. E.

204. Analysis of fibre mixtures. T. Szentpály, R. Szentpály and A. Stark (Res. Inst. of the Textile Ind., Budapest). *Faserforsch. u. Textiltech.*, 1960, **11** (4), 189–195.—Methods are described for the quant. analysis of mixtures containing two or more of the following types of fibre, *viz*, wool, regenerated protein, cotton, cellulose rayon, polyamide, polyester and poly(vinyl cyanide), by determining the losses in weight caused by treating the sample with a selective solvent for a single type of fibre, or with a number of different solvents in succession. Full details are given of these solvents, and tables show their applicability to all possible 2 to 4-component mixtures of these types of fibres.

H. L. WHITEHEAD

205. Reflectance spectrophotometric determination of soluble iron in delustered acrylic fibres. M. E. Gibson, jun., D. A. Hoes, J. T. Chesnutt and R. H. Heidner (Chemstrand Corp., Decatur, Ala., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 639–642.—The sample is dissolved in dimethylformamide, the Fe is reduced with hydroxyammonium chloride, and a coloured complex is prepared with a soln. of 1,10-phenanthroline in dimethylformamide. The presence of TiO$_2$ in the sample renders the soln. turbid or translucent, so that transmittance measurements are inapplicable, and the colour is measured by reflectance at 510 and 610 mμ, with a magnesium oxide reference standard, and corrected for a blank and for the Fe in the reagents. The difference between the corrected readings at 510 and 610 mμ is referred to a calibration curve. The standard deviation (8 determinations) for 2 p.p.m. of Fe was ±0·20. G. P. COOK

206. Determination of the fluorine content of high polymers. E. Schröder and U. Waurick (Inst. f. Chem. Tech. der Plaste, Leipzig). *Plaste u. Kautsch.*, 1960, **7** (1), 9–11.—The polymer is heated with sodium in a nickel crucible, the excess of sodium is destroyed, and the aq. solution of the product is passed through an ion-exchange column. The F content of the percolate is determined by titration with 0·1 N alkali. Any Cl present may be determined argentimetrically.   L. A. O'Neill

207. Determination of monomer in polystyrene and finished products. J. Eisenbrand and H. W. Eich (Chem. Untersuchungsamt f.d. Saarland, Saarbrücken). *Z. anal. Chem.*, 1960, **175** (1), 4–9 (in German).—The sample (0·1 g) is dissolved in 50 ml of $CHCl_3$, and 10 ml of the soln. is added slowly from a burette to a flask containing 70 to 80 ml of methanol. The soln. is shaken, diluted to 100 ml with methanol and again shaken, then set aside for 16 hr. The dissolved monomer is filtered off from the pptd. polymer and the extinction of the clear soln. is measured at 245 mμ. The calibration curve, prepared from known soln. of freshly distilled styrene dissolved in methanol, is linear. The max. error is 0·03%.   B. B. Bauminger

208. Analysis of simple alkyd resins. E. Schröder and K. Thinius (Inst. für Chem. Tech. der Plaste, Leipzig). *Dtsch. Farben-Z.*, 1960, **14** (4), 144–148; (5), 189–193.—Fatty acids recovered from alkyd resins have been examined by reversed-phase chromatography on paper impregnated with liquid paraffin, and acetic acid (90%, saturated with paraffin oil) as the mobile phase. Alkyds prepared from drying and non-drying oils were examined, but in several cases the spots obtained from the recovered fatty acids were different from those of the acids in the parent oil, and the method could not be considered as generally reliable for identifying the oil component of an alkyd. Procedures for the determination of dibasic acids, fatty acids and unsaponifiable matter, which are carried out after saponification with Na ethoxide, are given. The most convenient method for the determination of polyhydric alcohol involves aminolysis with ethanolamine and separation and oxidation of the alcohol.   L. A. O'Neill

209. Identification of the glycol components of unsaturated polyester resins by paper chromatography. H. Rejhová and W. Ulbrich (Forschungsinst. f. Kunstharze, Pardubice, Czechoslovakia). *Plaste u. Kautsch.*, 1959, **6** (11), 539–540.—The resin is saponified with ethanolic KOH, the K salts of the dibasic acids are filtered off, the alkali is neutralised with CO_2, the K_2CO_3 and the ethanol are removed, and the glycols are examined by ascending paper chromatography, with n-butanol saturated with H_2O as the mobile phase; the spots are revealed with ammoniacal $AgNO_3$. Results obtained on polyesters agreed with those obtained by conversion of the glycol into a 3,5-dinitrobenzoate or diphenylcarbamate derivative for identification.   L. A. O'Neill

210. Determination of water in pigment pastes and printing ink colours. F. Öhme (Inst. zur Entwicklung Analysenmethoden, Weilheim/Obb, Germany). *Farbe u. Lack*, 1960, **66** (3), 142–145.—The material is extracted with dioxan; the dielectric constant of the dioxan extract is determined before and after passage through a column of a drying agent, and the difference is a measure of the H_2O present.   L. A. O'Neill

211. Volumetric analysis with complexans in the paint laboratory. O. Borchert. *Plaste u Kautsch.*, 1959, **6** (11), 562–568.—Applications of complexometric methods to the analysis of paint compositions, *e.g.*, the determination of metals present in pigments and in driers, are reviewed.   L. A. O'Neill

212. Determination of chlorendic acid in fire-retardant paint. G. G. Esposito and M. H. Swann (Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 680–681.—The sample is dissolved in benzene and saponified with KOH in isopropyl alcohol. The resulting dipotassium salt of chlorendic acid (3,6-endodichloromethylene-3,4,5,6-tetrachloro-Δ⁴-tetrahydrophthalic acid) (I) is filtered off and dissolved in H_2O; the free acid is liberated by addition of H_2SO_4 and extracted into ethyl ether. The ether is washed with water until free from other acids and the **I** is determined by titration in ethanol with KOH, with *m*-cresol purple as indicator. By this method 47·0 and 47·3% of **I** were recovered from a sample known to contain 47·2%.   G. P. Cook

213. Rapid identification of elastomers and their additives. B. Cleverley and R. Herrmann (D.S.I.R., Wellington, New Zealand). *J. Appl. Chem.*, 1960, **10** (5), 192–195.—Elastomers can be identified from the i.r. spectra of their pyrolysates if they are pyrolysed under controlled conditions and interfering substances removed by fractional distillation. Identification of additives separated by fractional sublimation or distillation is possible in many cases. The spectra of the pyrolysates of nine common elastomers are illustrated.   G. P. Cook

214. Quantitative analysis of rubber ingredients. V. Simple combustion tube for the semi-micro determination of sulphur in vulcanisates. K. Hummel and H. H. Bertram (Kautschukinst., Tech. Hochschule, Hannover, Germany). *Kautsch. u. Gummi*, 1960, **13** (5), WT132–134.—The combustion tube described is packed with a mixture of vanadium pentoxide and quartz (2:1) and, after insertion of the sample, heated to 800° to 1000°. The resulting H_2SO_4 is absorbed in H_2O_2 and titrated complexometrically (*cf.* Bauminger, *Anal. Abstr.*, 1957, **4**, 3389). The rubber-combined sulphur and sulphur in some inorganic compounds, *e.g.*, ZnS, can be determined by this combustion method. Results showing the accuracy of the method for natural rubber vulcanisates of known sulphur contents are given. The sulphur in alkaline-earth-metal sulphates is determined by adding 1 g of a catalyst mixture of vanadium pentoxide and zinc oxide (4:1) to the sample in the boat (*cf.* Bauminger, *Analyst*, 1956, **81**, 12).   S. R. P.

215. Practical identification of some condensed tannins in leather by chromatographic and photometric methods. D. G. Roux (Leath. Ind. Res. Inst., Grahamstown, S. Africa). *J. Amer. Leath. Chem. Ass.*, 1960, **55** (5), 272–284.—The leather sample is powdered and extracted with acetone - water (1:1) for 2 hr. The soln. is filtered and the filtrate evaporated to dryness *in vacuo*. The extract (3 to 10 mg) is dissolved in 3 to 5 drops of methanol and applied to Whatman No. 1 paper, and developed with water-satd. s-butyl alcohol or n-butanol - acetic acid - water (4:1:5) in one direction and then with 2% acetic acid in the other direction. The paper may be sprayed with diazotised benzidine reagent, vanillin - toluene-*p*-sulphonic acid reagent

ammoniacal $AgNO_3$ soln., or $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. The anthocyanidins are recognised by their colorations and by their light-absorption characteristics.

<div align="right">C. A. SLATER</div>

216. Paper electrophoresis and chemical analysis of extracts of steerhide corium and of whole steerhide. C. Deasy (Tanners' Counc. Lab., Univ. of Cincinnati, Ohio, U.S.A.). *J. Amer. Leath. Chem. Ass.*, 1960, **55** (5), 258–271.—Methods of prep. of steerhide-corium extracts have been described elsewhere (*Ibid.*, 1959, **54**, 503). Whole-steerhide extracts are made from steerhide from a freshly slaughtered steer after freezing for one week. The hide is cut into squares and extracted successively with 5% NaCl soln., 0·1 M citrate buffer (pH 3·6), 0·1 M Na_2HPO_4, half-saturated $Ca(OH)_2$ soln. and 10% $CaCl_2$ soln. Extracts are concentrated by dialysis. Glycoproteins are determined by a modified Grönwall and Köiw procedure (*Scand. J. Clin. Lab. Invest.*, 1952, **4**, 244). Results show that corium extracts contain little or no glycoprotein, whilst whole hide contains large quantities.

<div align="right">C. A. SLATER</div>

See also Abstracts—**18**, ^2H in org. compounds. **22**, Li in mineral-oil dispersions. **122**, Cl in tetra-n-butyl titanate. **173**, Analysis of dithiocarbamates. **234**, Detection of glucose. **267**, Chromatography of triterpenoids. **283**, Determination of deoxysugars. **308**, Determination of amylose and amylopectin. **334**, Aldehydes in air. **335**, Toluene and styrene in air. **360**, Recording analyser for org. acids. **361**, Fractionation of org. acids. **372**, Chromatographic determination of ^{14}C-labelled salicylic acid. **377**, Response of thermal-conductivity detectors to org. compounds. **379**, Gas chromatography of alcohols.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

217. Desalting of urine by electrodialysis. T. Wood (Biochem. Dept., Univ. of Sydney, New South Wales). *Nature*, 1960, **186**, 634–635.—An electrodialyser described by Wood (*Biochem. J.*, 1956, **62**, 611) for the desalting of tissue extracts has been modified and used by Smith ("Chromatographic Techniques, Clinical and Biochemical Applications," Heinemann, London, 1958) for desalting urine, but considerable losses of amino acids were reported. It is now recommended that a "desalting curve" be plotted (cell resistance *vs.* time) so that the electrodialysis can be stopped at a point at which most of the salt has been removed, but little or no amino-acid lost.

<div align="right">G. S. ROBERTS</div>

218. Effects of haemolysis on serum electrolyte values. A. Mather and N. R. Mackie (Res. Dept., Memorial Hosp., Wilmington, Del., U.S.A.). *Clin. Chem.*, 1960, **6** (3), 223–227.—Correction of the potassium and phosphate found in freshly separated haemolysed sera for the contribution of the cells may be made with reasonable accuracy if the serum haemoglobin is determined. The observed value for K (milli-equiv. per litre) − 3·3 × serum haemoglobin (g per 100 ml) gives the corrected value for K. The correction for phosphate is ordinarily not significant, but may be made by deducting one third of the serum haemoglobin from the mg of P per 100 ml found.

<div align="right">H. F. W. KIRKPATRICK</div>

219. Automatic method for the determination of carbon dioxide in blood plasma. L. T. Skaggs, jun. (Dept. of Medicine and Surgery, Veterans Admin. Hosp., Cleveland, Ohio). *Amer. J. Clin. Path.*, 1960, **33** (2), 181–185.—An AutoAnalyser is employed to measure the colour change produced in a phenolphthalein soln. by CO_2.

<div align="right">D. E. EVANS</div>

220. Contemporary carbon-14. The *p*-cymene method. F. N. Hayes, E. Hansbury and V. N. Kerr (Los Alamos Sci. Lab., Univ. of California, N. Mex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 617–620.—Carbon-14 in nature is being investigated on a world-wide scale and details are given of the sampling, chemical conversions and liquid scintillation counting procedures used, special attention being paid to the use of *p*-cymene from natural terpene sources for the liquid scintillation method.

<div align="right">K. A. PROCTOR</div>

221. Combined potentiometric determination of serum bicarbonate and chloride. M. Rockenmacher (Clin. Lab., Dept. of Pathology, Los Angeles County Harbor General Hospital, Torrance, Calif.). *Amer. J. Clin. Path.*, 1960, **33** (4), 349–354.—As little as 0·2 ml of serum is sufficient for this titrimetric method, the results of which compare well with those of the Van Slyke and Whitehorn methods.

<div align="right">D. E. EVANS</div>

222. Determination of sodium and potassium in biological fluids with the dual-channel ultra-micro flame photometer. P. A. Bott (Dept. of Physiol. Chem., Woman's Med. Coll. of Pennsylvania, Philadelphia, U.S.A.). *Anal. Biochem.*, 1960, **1** (1), 17–22.—A modification of the instrument designed by Ramsay *et al.* (*J. Exp. Biol.*, 1953, **30**, 1) is described. As little as 2 $\mu\mu$moles of Na and K can be determined simultaneously in 0·2 μl of sample.

<div align="right">H. F. W. KIRKPATRICK</div>

223. Analysis for potassium, sodium, chloride and water in a 2-μl sample of extracellular fluid. A. Ames, III, and F. B. Nesbett (Harvard Medical School, Boston, Mass.). *Anal. Biochem.*, 1960, **1** (1), 1–7.—The samples (2 to 8 μl) are weighed into small stoppered tubes, dried for 4 hr. at 105°, and re-weighed to give the water content. The extracting soln. (0·220 millimole of $AgNO_3$ and 5 millimoles of Li_2SO_4 per litre of 0·1 N HNO_3) is added in a vol. corresponding to 1000 times the water content of the sample. The Cl^- are determined by measuring the residual Ag^+ spectrophotometrically with the rhodanine reagent of Lowry *et al.* (*J. Biol. Chem.*, 1954, **207**, 1). The K and Na are determined by flame photometry in a specially designed photometer, the Li^+ in the extraction soln. serving as internal standard. The accuracy and reproducibility are better than 2%.

<div align="right">R. A. BRENAN</div>

224. Determination of free magnesium ions in body fluids. Improved methods for free calcium ions, total calcium and total magnesium. M. Walser (The Johns Hopkins Univ. Sch. of Med., Baltimore, Md., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 711–717.—The colour change on the addition of Eriochrome black T to a protein-free sample of body fluid, buffered with tri(hydroxymethyl)-methylamine, gives an accurate estimate of the free Mg^{2+} concn. At constant pH and constant

ionic strength, the reciprocal of the extinction difference is a linear function of the reciprocal of the Mg^{2+} concn. Heavy-metal ions must be removed before the analysis; other cations present in body fluids do not interfere. A similar procedure is used for free Ca^{2+}, with murexide as indicator. The sum of total Ca and total Mg is determined by titration with EDTA (disodium salt) soln. at pH 10, with Eriochrome black T as indicator. Total Ca is determined by titration in strongly alkaline soln., with Cal-Red as indicator (Bachra *et al.*, *Clin. Chem.*, 1958, **4**, 107).   A. R. ROGERS

225. Micro calcium method accurate in the presence of magnesium and phosphate. T. B. Coolidge (Dept. of Biochem., Univ., Chicago, Ill.). *Anal. Biochem.*, 1960, **1** (1), 93–95.—Serum (0·5 ml) is pipetted into a Pyrex-glass tube (100 mm × 13 mm) and dried at 110°. A mixture (0·2 ml) of equal parts of HNO_3 and 70% $HClO_3$ is added and the tube is placed for 5 min. at a depth of 20 mm, for 15 min. at a depth of 40 mm, and for 30 min. at a depth of 98 mm in an aluminium block heated to 180° and provided with suitable holes and plugs to give the required depths. To the ash is added water (0·5 ml), fresh aq. dilution (1:5) of stock 2% calcein soln. in 0·25 N NaOH (2 drops), and 2 N CO_2-free NaOH (0·4 ml). About 0·5 ml (measured to 0·1% accuracy) of 0·004 M EDTA, prepared by dilution of 0·04 M EDTA in 0·081 N NaOH, is added from a syringe pipette, and the mixture is titrated with 0·100 M $CaCl_2$ from a micro-burette, with stirring by filtered air.   R. A. BRENAN

226. Photo-electric complexan titration of calcium and magnesium in serum. M. Kortüm (Inst. f. Phys. Chem., Univ. Tübingen, Germany). *Klin. Wochschr.*, 1960, **38** (9), 452–457.—The following procedures are proposed. *Calcium*—Mix in a cell 0·0025 M EDTA (disodium salt) (1·0 ml), N NaOH (1·5 ml), 5% Na_2S soln. (0·15 ml), serum (1·0 ml), 0·1% aq. murexide soln. (0·15 ml) and H_2O (3·5 ml) in that order. Place the cell in the absorptiometer and set the extinction to 0·1 to 0·3 at 490 mμ. Add 0·0025 M Ca soln. dropwise from a micro-burette, taking readings after each addition. The extinction at first remains approx. constant, then rises. The end-point is reached when the addition of a drop of titrant produces a permanent increase in the extinction. Then mg of Ca per 100 ml of serum = (2·0 − no. of ml of Ca soln. added) × 10. *Magnesium*—Mix in a cell 0·0025 M EDTA (disodium salt) (2·0 ml), 2·5% aq. NH_3 soln. (1·5 ml), 5% Na_2S soln. (0·2 ml), serum (1·0 ml), 0·1% aq. catechol violet soln. (0·15 ml) and H_2O (3·5 ml) in that order. After 2 min., place the cell in the absorptiometer and set the extinction to approx. 0·8 at 620 mμ. Titrate dropwise as described above. Then mg of Mg per 100 ml of serum = (no. of ml of Ca soln. added in the murexide titration − ml of Ca soln. added in the catechol violet titration) × 6·1.   H. F. W. KIRKPATRICK

227. Complexometric determination of calcium and magnesium in urine. S. I. Pekhtereva (Lenin Inst. of Med., Moscow). *Lab. Delo*, 1960, **6** (3), 17–20.—*Procedure*—To 5 ml of urine add 5 ml of 0·2 N Na oxalate, mix and set aside for 1 hr. at room temp., then filter. To one flask (*a*) add 1 ml of urine and 1 ml of water, and to another (*b*) 2 ml of filtrate. Then to each flask add 1 ml of 0·03% ethanolic methyl red soln., 1 ml of 0·1% ethanolic Chromogen black ET-00 (C.I. Mordant Black 11) soln. and 0·5 ml of buffer soln. (20 g of NH_4Cl and

10 ml of 25% aq. NH_3 in 1 litre of water). Titrate with 0·01 N EDTA (disodium salt) dropwise, with vigorous shaking after each addition, to a colour change from violet - red to green. The amount of EDTA used in (*a*) corresponds to the sum of Ca and Mg and the amount used in (*b*) to Mg alone; Na_2HPO_4 does not interfere, and the errors are ≯ 3%.   A. BURWOOD-SMITH

228. Wet digestion of plant material gives low boron values. J. T. Hatcher (Soil and Water Conservation Res. Div., U.S. Dept. of Agric., Riverside, Calif.). *Anal. Chem.*, 1960, **32** (6), 726.—Results on 12 samples show that the wet-digestion procedure (*cf.* U.S. Dept. Agric., *Agric. Handb.*, 1954, **60**, 129, 135) is not satisfactory. Serious loss of boric acid occurs by volatilisation from the acid soln. during the digestion.   A. R. ROGERS

229. Determination of silicic acid in biological material. H. Baumann (Silikose-Forschungsinst., Bergbau-Berufsgenossenschaft, Bochum, Germany). *Hoppe-Seyl. Z.*, 1960, **319**, 38–51.—A colorimetric method is described for the determination of silicic acid by the reduction of the molybdosilicate complex with a mixture of 1-amino-2-naphthol-4-sulphonic acid and ascorbic acid. Other reducing substances, PO_4^{3-} and Fe do not interfere. Protein is removed by combustion or ultrafiltration. A 3-ml sample is used and one operator can carry out 40 to 50 analyses in 8 hr. Much lower results have been obtained with this procedure than have been found with other techniques. Bovine blood or plasma has a silicic acid content of 2·2 μg per ml. This is not bound to protein or other substances of high mol. wt.
F. POWELL

230. Micro-method for estimating inorganic phosphorus in serum or plasma. J. F. Johnston (Biochem. Dept., Royal Hospital for Sick Children, Glasgow). *J. Med. Lab. Technol.*, 1960, **17** (1), 25–27.—This is a modification, for application to micro amounts, of the method of Bell and Doisy (*J. Biol. Chem.*, 1920, **44**, 55).   R. A. BRENAN

231. Determination of phosphorus, radio-phosphorus and chromic oxide in faeces with a solution obtained by perchloric acid oxidation. Ichiro Yoshihara and Akio Sugisaki (Agric. Fac., Niigata Univ., Japan). *J. Agric. Chem. Soc. Japan*, 1959, **33** (1), 27–31.—Chromic oxide and ^{32}P were used as tracers for the study of absorption and excretion of P in rabbits, and the separate determination of Cr, ^{32}P and total P in faeces was carried out as follows. The sample (≃1·2 g) is heated with 60% $HClO_4$ soln. (20 ml) for 30 min., then cooled and treated with H_2O_3 (10 drops). The soln. is heated again, mixed with water (80 ml), filtered and diluted to 200 ml. This soln. is used for the determination of total P by Allen's method (*Biochem. J.*, 1940, **34**, 858), of ^{32}P by counting after pptn. with magnesia mixture, and of Cr by colorimetry.   S. NATORI

232. Determination of chromic oxide and barium sulphate used as a trace material for the movement of intestinal contents. Ichiro Yoshihara (Agric. Fac., Niigata Univ., Japan). *J. Agric. Chem. Soc. Japan*, 1959, **33** (2), 138–141.—The conditions for the separate determination of $BaSO_4$ (**I**) and Cr_2O_3 (**II**) were examined, and the determination of **I** by the isotope dilution method and **II** by titration was improved and applied to the determination of **I** and **II** in faeces.   S. NATORI

233. Spectrographic determination of zinc in plant material ash using the 2138·6-Å spectrum line. A. Strasheim and D. J. Eve (Nat. Phys. Res. Lab., S. African Council for Sci. & Ind. Res., Pretoria). *J. S. Afr. Chem. Inst.*, 1960, **13** (1), 7–12.—In the method described, Ilford "Q1" u.v.-sensitive plates were used, and modifications were made to earlier procedures to minimise errors. A Stallwood-type air jet was used round the arc to overcome the difficulty of self-absorption. A constant-current source (Van der Walt, *Rev. Sci. Instrum.*, 1956, **27**, 157) minimised variations in emission. The sample was diluted (1:9) with a buffer mixture of 10% of K_2SO_4 and 40% of $CaCO_3$ in graphite to reduce the influence of variation in the main constituents of the sample. The method is fairly rapid, apart from the ashing, and is sufficiently accurate for nutritional studies on trace elements. The error is $\simeq 5.4\%$. 　　　　　　　　　　　A. M. SPRATT

234. Specific detection of glucose on paper chromatograms. M. R. J. Salton (Dept. of Bacteriology, Univ. Calif., Berkeley, U.S.A.). *Nature*, 1960, **186**, 966–967.—Glucose may be detected and distinguished from other monosaccharides by spraying the dried chromatogram with a glucose oxidase indicator reagent ("Glucostat," Worthington Biochemical Corp.). When acid solvents, such as n-butanol-acetic acid, have been used, the dried paper should be steamed for 5 to 10 min. before spraying. Glucose develops a pink-brown spot at room temp., and may be detected in amounts of 10 to 20 mμg, or less. The coloured complex can not be eluted for quantitative determination. After detection of glucose, other monosaccharides may be detected by a further spraying with aniline phthalate. 　　　　　　　　　　　D. G. MOSS

235. Determination of alcohol in blood by gas chromatography. B. Chundela and J. Janák (Lab. Toxicol. and Forensic Med., Charles' Univ., Prague). *Soudní Lékařství*, 1959, **4** (10), 145–150.— A 2% aq. soln. of n-butanol (0·25 ml) is added to the sample (1 ml) and 0·1 ml of the mixture is placed by means of a syringe on the column. The column consists of 25% of diglycerol on Sterchamol (0·2 to 0·4 mm mesh) activated for 3 hr. at 400°. A Griffin and George apparatus, type MK II, is used, and the n-butanol serves as an internal standard. The sensitivity is 0·1%. 　　　J. ZÝKA

236. Identification of corticosterone sulphate in human urine after administration of corticosterone. J. R. Pasqualini. *Compt. Rend.*, 1960, **250** (23), 3892–3893.—It is shown that sulphate ester is excreted within 36 hr. by a normal subject given corticosterone. The ester is separated by two chromatograms on Al_2O_3 and identified by chromatography in four solvent systems, synthetic corticosterone 21-sulphate being run in parallel. The parent alcohol is similarly identified, after hydrolysis with sulphatase (from *Helix pomatia*), by chromatography in three solvent systems, with corticosterone run in parallel. 　　　　J. P. STERN

237. Determination of starch in leaves and other parts of plants. Kh. N. Pochinok. *Nauch. Trud. Ukr. Inst. Fiziol. Rast.*, 1959, (20), 59–62; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (9), Abstr. No. 11,826.—Volumetric and colorimetric methods have been developed. Dissolve the starch by heating with 80% $Ca(NO_3)_2$ soln. and separate it from other substances by pptn. with iodine. The resulting ppt. of starch is quant. oxidised in H_2SO_4 medium with

$K_2Cr_2O_7$ to CO_2 and water, which enables it to be determined by a simple volumetric method. From 1 to 10 mg of starch can be determined, with an accuracy of $\pm 1\%$. The starch pptd. with iodine can also be determined colorimetrically after dissolving the starch-iodine complex in alkali; 0·5 to 5 mg of starch can be determined, with an accuracy of $\pm 5\%$. 　　　　　　C. D. KOPKIN

238. Micro-method for the determination of laevulic acid. Nai-Siang Jiang and D. P. Groth (Dept. of Pharmacol. and Biochem., Emory Univ., Atlanta, Ga.). *Anal. Biochem.*, 1960, **1** (1), 40–43.— The dinitrophenylhydrazone is formed in benzene soln. in the presence of H_2SO_4, and extracted with 0·5 N NaOH. The colour is measured at 540 mμ. The method can be used to determine 2·5 to 25 μg of laevulic acid. 　　　H. F. W. KIRKPATRICK

239. New technique for the determination of galacturonic acid in cerebrospinal fluid. L. Buscarini (Ist. di Clin. Med. Generale e Terapia Med., Univ. Pavia, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1960, **36** (11), 564–567.—The method is based on the application of the Dische reaction (*J. Biol. Chem.*, 1947, **167**, 189) to the determination of galacturonic acid separated from protein by pptn. with protamine sulphate. *Procedure*—To cerebrospinal fluid (1 ml) add water (4 ml) and a 1% soln. of protamine sulphate (0·3 ml), shake and centrifuge. Dissolve the ppt. in 2 M acetate buffer (pH 5) (3 ml). To the sample soln. (1 ml), to a standard soln. containing 5 mg of glucuronic acid per 100 ml (1 ml), and to a buffer blank (1 ml) add H_2SO_4 (6 ml), drop by drop, with cooling. Heat at 100° for 20 min., allow to cool and measure the developed (non-specific) colour at 535 mμ. To each soln. add a 0·1% ethanolic soln. of carbazole (0·2 ml), set aside for 2 hr. and measure the colour developed at 535 mμ. The increase in colour in the sample soln. is calculated to glucuronic acid; glucuronic acid found × 2·32 = galacturonic acid. Results obtained for normal human subjects were 177 ± 8.70 mg of galacturonic acid per 100 ml. 　　　E. C. APLING

240. Enzymatic determination of hyaluronic acid. H. Greiling, T. Gunther and T. Eberhard (Physiol.-Chem. Inst., Freien Univ., Berlin). *Hoppe-Seyl. Z.*, 1960, **319**, 161–166.—The u.v. absorption of the disaccharide formed by the action of bacterial hyaluronidase may be used to determine hyaluronic acid. The material to be tested is incubated for 6 hr. at pH 6·4 with a preparation of hyaluronidase from *Staphylococcus aureus* and the extinction is read at 230 mμ. Beer's law is obeyed over the range 20 to 700 μg of hyaluronic acid. Above a mol. wt. of 4000 the method is not influenced by the degree of polymerisation of the hyaluronic acid. The mean error is $\pm 2\%$. 　　　　F. POWELL

241. Positive identification of flavanone aglycones by paper chromatography of their alkaline degradation products. W. J. Dunlap and S. H. Wender (Chem. Dept., Univ. of Oklahoma, Norman, U.S.A.). *J. Chromatography*, 1960, **3** (5), 505–507 (in English).—Flavanones are degraded by heating the aglycone under reflux with a little 30% aq. KOH soln. for 2·5 hr. The mixture is then made acid (pH 4) with dil. H_2SO_4 and extracted with ethyl ether, and the extract is chromatographed. The solvents used are n-butanol-ethanol-aq. NH_3/ammonium carbonate buffer (40:11:19) and a modification prepared from 1 part of nitromethane

with 2 parts of n-butanol saturated with the ammoniacal buffer. Diazotised sulphanilic acid and *p*-nitroaniline spray reagents are used for the detection of hydroxycinnamic acids and phloroglucinol, but for cinnamic acids possessing only methoxy substituents, and which do not therefore couple, buffered methyl red or 2,6-dichlorophenolindophenol reagents are useful. The method has been successfully applied to the identification of unknown compounds obtained by hydrolysing the glycosides from citrus fruits. S. M. MARSH

242. **Isolation and identification of lysolecithin from lipid extracts of normal human serum.** E. Gjone, J. F. Berry and D. A. Turner (Biochem. Res. Div., Dept. Med., Sinai Hosp., Baltimore, Md., U.S.A.). *J. Lipid Res.*, 1959, **1**, 66–71.—Lipids were extracted from 15 to 20 ml of serum with 25 vol. of $CHCl_3$ - methanol (2:1). The lipid extract was washed with water and the solvent removed. A soln. of the lipids in $CHCl_3$ was subjected to chromatography on silicic acid. Ethyl ether was used to elute the non-phospholipids, then the column was eluted with $CHCl_3$ - methanol. The phospholipids eluted successively were cephalin, lecithin, sphingomyelin and lysolecithin. The identity of each component was confirmed by paper chromatography, chemical analysis and infra-red absorption spectra. Lysolecithin P represented $\simeq 9\%$ of the total lipid P of serum. NUTR. ABSTR. REV.

243. **Determination of serum glycerides.** L. A. Carlson (King Gustav V Res. Inst., Stockholm). *Acta Soc. Med. Uppsaliensis*, 1959, **64**, 208–213.— Lipids are extracted with $CHCl_3$ - methanol (2:1) from 1 ml of serum and the extract is washed with NaCl soln. The remaining $CHCl_3$ soln. is passed through the column of silicic acid on which the phospholipids are selectively retained. A portion of the $CHCl_3$ percolate is then saponified with ethanolic KOH soln., dil. H_2SO_4 is added and fatty acids and unsaponifiable matter are extracted with ethyl ether; the residual aq. layer is used for the determination of glycerol by the method of Carlson and Wadström (*Anal. Abstr.*, 1959, **6**, 4083).
NUTR. ABSTR. REV.

244. **Seromucoid determination: variables and modifications.** T. M. Asher and G. R. Cooper (Communicable Diseases Centre, Public Health Service, Atlanta, Ga., U.S.A.). *Clin. Chem.*, 1960, **6** (3), 189–198.—The method of Winzler ("Methods of Biochemical Analysis," 2nd Ed., New York, 1955, p. 279) is investigated. A modified technique is described for making the test more sensitive. H. F. W. KIRKPATRICK

245. **Enzymatic spectrophotometric method for the determination of uric acid.** L. Liddle, J. E. Seegmiller and L. Laster (Nat. Inst. Health, Bethesda, Md., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (6), 903–913.—A detailed description of the uricase method (*cf.* Feichtmeir and Wrenn, *Amer. J. Clin. Path.*, 1955, **25**, 833) and an evaluation of the method in 4000 analyses during clinical studies of uric acid metabolism are given. The specificity, reproducibility and accuracy, and results in relation to those given by other methods, are discussed.
W. H. C. SHAW

246. **Effect of orally administered quinine and quinidine on apparent values for urinary catecholamines.** S. M. Sax, H. E. Waxman, J. H.

Aarons and H. J. Lynch (Dept. of Path., W. Pennsylvania Hosp., Pittsburgh, U.S.A.). *Clin. Chem.*, 1960, **6** (2), 168–175.—Oral administration of quinine or quinidine yields urinary derivatives, possibly carbostyrils, that cause an apparent increase in urinary catecholamines.
H. F. W. KIRKPATRICK

247. **Determination of creatinine in small quantities of plasma.** E. Martinez and P. D. Doolan (Clin. Invest. Centre, U.S. Naval Hosp., Oakland, Calif.). *Clin. Chem.*, 1960, **6** (3), 233–242.—A modification of the method of Van Pilsum *et al.* (*J. Biol. Chem.*, 1956, **222**, 225) is described and is recommended when accuracy is the primary requirement. For routine use a modification of the alkaline picrate method is proposed.
H. F. W. KIRKPATRICK

248. **Determination of choline in blood by antimony chloride in ethereal solution.** H. Wachsmuth and R. Denissen (Lab. de Chim. et de Biochim., Hôpital Stuivenberg, Anvers). *J. Pharm. Belg.*, 1959, **14** (3–4), 141–144.—*Procedure*—Treat 3 to 4 ml of blood with Bloor's mixture. Evaporate the alcoholic soln. and re-dissolve the residue in 1 ml of light petroleum. Add 8 ml of acetone and 2 to 3 drops of satd. ethanolic $MgCl_2$ soln. to precipitate the phosphatides. Treat the ppt. with satd. $Ba(OH)_2$ soln. and heat on a boiling-water bath for 2 hr. Acidify with N H_2SO_4, then make alkaline with 0·05 N NaOH, and neutralise with 0·1 N HCl to the phenolphthalein end-point. Centrifuge, evaporate an aliquot to dryness at $\div 40°$, re-dissolve the residue in ethanol and reduce the vol. to 0·2 to 0·3 ml in a centrifuge tube. Add SbI_3 soln. (make a suspension of 0·2 g of SbI_3 in 5 ml of ethanol; after 3 min. add 15 ml of peroxide-free ethyl ether and filter), dropwise, and make up to 7 to 8 ml with ether. Centrifuge the yellow ppt. in a closed tube, decant off the ether, and wash the ppt. twice with ether. Dissolve the ppt. in 1 ml of 30% NaOH soln., dilute to $\simeq 100$ ml, acidify slightly with H_3PO_4 and add 20 ml of bromine water. Remove the excess of Br by boiling, add 2 to 3 drops of 5% Na salicylate soln., acidify with H_3PO_4, and add solid KI. Titrate the liberated iodine with 0·01 N $Na_2S_2O_3$ (1 ml ≡ 0·0871 mg of choline chloride). Results are compared with those obtained with ammonium reineckate. R. A. HENDEY

249. **Sensitivity of the fluorescence test for amino-acids.** J. Opieńska-Blauth, M. Sanecka and M. Chareziński (Biochem. Dept., Med. Acad., Lublin, Poland). *J. Chromatography*, 1960, **3** (5), 415–424 (in English).—The sensitivities of the ordinary u.v. fluorescence test, the sensitised u.v. test (sensitising agent Na 1,2-naphthaquinone-4-sulphonate soln.) and the ninhydrin and isatin tests have been compared for 25 amino-acids. The sensitised u.v. test was ten times as sensitive as the ordinary u.v. test, except with asparagine, lysine and tryptophan. The best results are obtained by heating the chromatogram at 110° to 120° for 10 min. The effect of developing the chromatogram with phenol - water (7:3) and n-propanol - water (7:3) was to reduce the sensitivity owing to losses during migration, particularly for amino-acids with high R_F values. Amino-acids in biological fluids such as urine and blood serum can be identified by applying the u.v. and ninhydrin tests successively, provided that the amount of sample applied is not less than 100 μl. S. M. MARSH

250. Determination of amino-acids with dithionite-reduced ninhydrin. H. Stegemann (Max-Planck-Gesellschaft, Göttingen, Germany). *Hoppe-Seyl. Z.*, 1960, **319**, 102–109.—A procedure is described for the determination of amino nitrogen by means of ninhydrin partially reduced with dithionite. The presence of NaF and a high concn. of acetate buffer in the reagent make it possible to use soln. containing air. The optimum range is 1 to 3 μg of N per ml. The accuracy in this range is $\pm 2\%$; Beer's law is obeyed up to 10 μg. The colour is stable for 1 hr. and it is claimed that it is possible to analyse more than 1000 samples per day.

F. POWELL

251. Determination of neutral amino-acids by partition chromatography on silica gel. Makoto Kandatsu and Hiroshi Naito (Fac. of Agric., Univ., Tokyo). *J. Agric. Chem. Soc. Japan*, 1959, **33** (3), 170–174.—The separation of N-acetamido-acids by chromatography on silica gel was investigated. The quality of the gel, *e.g.*, its purity, particle size and water content, especially of structural water, seemed to affect significantly the recovery of amino-acids. A mixture of acetylated amino-acids was eluted with ethyl acetate and then with $CHCl_3$-butanol containing an increasing proportion of butanol. The determination of valine in the presence of methionine was carried out after performic acid oxidation followed by acetylation and chromatography. Amino-acid components of casein and skeletal muscular protein of albino rats were determined by this method.

S. NATORI

252. Electrophoretic separation of some amino-acids from their copper complexes. M. Szwaj and M. Kański (Biochem. Dept., Med. Acad., Lublin, Poland). *J. Chromatography*, 1960, **3** (5), 425–430 (in English).—Complete separation of amino-acid - copper complexes into three groups, acid, neutral and alkaline, and separation of a given complex from the free amino-acid has been achieved by paper electrophoresis in a borate - NaOH buffer, pH 10, μ 0·05, at a potential gradient of 6·6 to 10·2 V per cm. The sample mixture is applied half way along the paper strip and electrophoresis is carried out for 1·5 hr. Ninhydrin is used as a locating agent for amino-acids and a 0·1% soln. of dithio-oxamide in methanol for the copper complexes. Results are detailed for 9 amino-acids and their complexes. The composition of the complex can be determined by precipitating the Cu with H_4S and chromatographing the liberated acid.

S. M. MARSH

253. Quantitative determination of 5-hydroxytryptophan, 5-hydroxytryptamine and 5-hydroxyindol-3-ylacetic acid by paper electrophoresis. A. Carcasona, F. Unterharnscheidt, J. Cervos-Navarro and W. Geller (Inst. f. Neuropathol., Univ. Bonn, Germany). *Klin. Wochschr.*, 1960, **38** (9), 457–460.—In barbitone buffer (pH 8·6), acetate buffer (pH 5·05) and phosphate buffer (pH 7·0), 5-hydroxytryptamine travels towards the cathode, 5-hydroxyindol-3-ylacetic acid towards the anode, and 5-hydroxytryptophan remains stationary. The three substances are therefore separable and can be determined by spraying the paper with p-dimethylaminobenzaldehyde soln. in n-butanol (1:4) and heating at 60° for 20 min. to produce a colour which is measured planimetrically on the paper at 540 mμ. The procedure is described.

H. F. W. KIRKPATRICK

254. Reduction of cystine as a basis for a simple amido-group determination for all proteins. H. Stegemann (Max-Planck-Gesellschaft, Göttingen). *Hoppe-Seyl. Z.*, 1959, **315**, 137–140.—Performic acid oxidation of cystine before the determination of amide nitrogen in proteins (*cf. Anal. Abstr.*, 1959, **6**, 4097) is not satisfactory for proteins rich in tryptophan. Reduction by Na thioglycollate or Na_2S is satisfactory for all proteins. The reduction of the cystine, alkaline hydrolysis of the amido-group, and diffusion of the NH_3 formed into H_2SO_4 are all carried out in one step. The actual working time is no longer than for a simple estimation of amido-groups, but 65 hr. is required for the complete diffusion of the NH_3.

F. POWELL

255. Chromatographic determination of cysteic acid. S. L. Bandemer and R. J. Evans (Dept. of Agric. Chem., Michigan State Univ., East Lansing, U.S.A.). *J. Chromatography*, 1960, **3** (5), 431–433 (in English).—The difficulties caused by variations in the volume of eluate fractions obtained in the determination of cysteic acid with ninhydrin, after its chromatographic separation on a resin column, have been overcome by the use of buffered soln. of 2 N NaOH saturated with Na citrate to produce the pH 5 required for maximum colour development. By using collector tubes calibrated to 10 ml and by constructing a standard cysteic acid curve the calculation of cysteic acid concn. has been simplified.

S. M. MARSH

256. Technique for the identification of peptides on paper by means of chlorine and o-tolidine. O. J. Schmid (Physiol.-Chem. Univ.-Inst., Hamburg, Germany). *J. Chromatography*, 1960, **3**(5), 499–500 (in German).—The paper chromatogram is sprayed with acetone - 30% ethanol (1:1) and suspended in freshly generated chlorine gas for 3 to 10 min. It is then transferred to a second vessel containing gaseous NH_3 and suspended there for 5 to 10 sec. The edge of the paper is tested by spraying with tolidine reagent (prepared from a satd. soln. of o-tolidine in 2% acetic acid with an equal vol. of 0·05 M KI) and if the background gives a blue colour the treatment with NH_3 is repeated. The whole chromatogram is then sprayed with the reagent. Amino-acids or peptides give blue - black or brown - yellow flecks, which can be photographed or photocopied before they fade.

S. M. MARSH

257. Combined agar gel - paper electrophoresis. B. Zak, F. Volini, J. Briski and L. A. Williams (Dept. of Pathology, Wayne State Univ. Coll. of Med., Detroit, Michigan). *Amer. J. Clin. Path.*, 1960, **33** (1), 75–82.—A method of agar-gel electrophoresis is described, whereby it is possible to evaluate the concn. of stained proteins in the separated zones; 25 to 45 min. is required for the complete separation of 5 or more protein zones. Resolution of serum proteins or haemoglobins in this open system is similar to that obtained with a closed system. Elution recoveries of separated serum proteins are almost identical with those of the Spinco procedure. Wet paper is laid on a glass plate and a boundary wall is formed of glass rods, to contain the molten agar. After electrophoresis, the gel - paper combination is dried and stained. Drying concentrates the protein bands down on to the surface of the paper, rather than into its substance, thus tending to give sharper definition of zones.

D. E. EVANS

258. Automatic determination of serum proteins. J. F. Failing, jun., M. W. Buckley and B. Zak (Dept. of Pathology, Wayne County General Hospital, Eloise, Michigan). *Amer. J. Clin. Path.*, 1960, **33** (1), 83–88.—Two absorptiometric methods are described. In the first, the biuret reaction is used for the determination of total serum proteins and, in the second, the selective reaction of methyl orange with serum albumin is used for the determination of this protein in the presence of other serum proteins. Concentration - transmittance curves are compiled for the AutoAnalyser, with standard protein solutions. In general the results compare well with those obtained by manual biuret-salt fractionation and manual methyl orange absorption. The values obtained by the automatic method show a slight compression of the range (high values less high, low values less low) compared with the values obtained manually. D. E. EVANS

259. Electrophoresis of soluble proteins in agar gel. A. T. Ilkov and T. Nikolov. *Voprosy Med. Khim.*, 1959, **5** (5), 388–392; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (7), Abstr. No. 8788.—The electrophoresis on agar of blood-serum proteins has been carried out with a potential gradient of 2·5 V per cm in a barbitone buffer of pH 8·6, with μ 0·05, and current 10 to 13 mA per sq. cm of cross-section of the agar. Under these conditions the serum proteins are separated by 12 to 13 cm in 4 to 4·5 hr. After the electrophoresis, the plate was twice immersed in a bath of 0·2 N acetic acid for 8 to 12 hr. and then dried in a thermostat at 37°. The dried plates were coloured for 5 hr. in a soln. of Amido black 10 B (C.I. Acid Black 1), and the background decolorised by immersion for 1 to 2 min. in 2% acetic acid containing 2·5% of glycerol. The dried plates were studied densitometrically. In the study by this method of human and animal blood serum, the presence of β_1- and β_2-globulins was noted, in addition to the peaks of albumin and α_1-, α_2-, β- and γ-globulins obtained by paper electrophoresis. In a large number of cases with human serum and in all cases with rabbit serum, an additional fraction, α_3-, was observed between the α_2- and β_1-fractions. Even more protein fractions were observed with dog sera. In some sera the β_2- and γ-globulin fractions were divided into subfractions. The described method has been used to determine the soluble proteins of tissues and the urease activity of the separate protein fractions of extracts of the mucous membrane of the stomach, urease preparations from soya, etc. C. D. KOPKIN

260. Separation of human serum lipoproteins by conventional descending and centrifugally accelerated paper chromatography. H. J. McDonald, L. J. Banaszak and J. Q. Kissane (Dept. of Biochem., Loyola Univ., Chicago, Ill.). *Anal. Biochem.*, 1960, **1** (1), 44–59.—The paper chromatography of lipids, including the centrifugally accelerated technique (McDonald *et al.*, *Anal. Chem.*, 1959, **31**, 825), is described. Correlation was established between lipoprotein fractions obtained by these methods and by ultracentrifugation. H. F. W. KIRKPATRICK

261. Separation of the α- and β-chains of globins by means of starch-gel electrophoresis. C. J. Muller (Dept. of Paediatrics, Univ. of Groningen, Holland). *Nature*, 1960, **186**, 643.—Human adult and foetal globin has been separated in chains by the starch-gel electrophoresis method of Smithies (*Biochem.*

J., 1955, **61**, 629). The α-chains of the two types of globin have equal mobilities, whereas the β-chains have different mobilities. G. S. ROBERTS

262. Improved method for the determination of haemoglobin A₂ by starch-gel electrophoresis. C. A. J. Goldberg and A. C. Ross (Wm. Pepper Lab. of Clin. Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chem.*, 1960, **6** (3), 254–262.—Modifications introduced into a method previously published (Goldberg, *Ibid.*, 1958, **4**, 484) improve the accuracy. H. F. W. KIRKPATRICK

263. A new electrophoretic medium and its application to the resolution of human haemoglobin variants. E. R. Huehns and A. O. Jakubovic (Dept. of Biochem., University College, London). *Nature*, 1960, **186**, 729–730.—The separation of human haemoglobin variants by gel-phase ion-exchange electrophoresis is investigated. Carboxymethylcellulose gel is the supporting medium and electrophoresis is carried out in a sodium phosphate buffer (0·17 to 0·04 M Na⁺). The best over-all separation, including the separation of haemoglobins A and F, is achieved with a Na⁺ concn. of 0·07 M. E. C. BUTTERWORTH

264. Far ultra-violet direct photometry applied to electrophoresis of nucleic acid. N. Ressler (Dept. of Med., Wayne County General Hospital, Eloise, Mich., U.S.A.). *Naturwissenschaften*, 1960, **47** (10), 228–229 (in English).—Zone electrophoresis is performed in the usual way in a film of transparent semi-fluid or thixotropic buffer layered on a quartz plate. At intervals during the course of the electrophoresis, light from a monochromator is passed through the film on to a photographic plate. Alternatively, a spectrophotometer photocell can be used. The migration of protein and nucleic acid can be observed at 200 mμ, then by changing the wavelength to 260 mμ, at which protein does not absorb, the nucleic acid alone can be observed. The method has been used to study the interaction between nucleic acid and protein during migration. R. A. BRENAN

265. Paper chromatography of nucleic acid derivatives. R. C. Ramos Barreto. *Rev. Quim. Ind., Rio de Janeiro*, 1960, **29**, 15–16.—Examples of paper-chromatographic separations of nucleic acid derivatives, including ³²P-labelled components, are described. L. A. O'NEILL

266. Determination of cholesterol with special reference to the use of toluene-p-sulphonic acid. L. A. Wright, D. B. Tonks and R. H. Allen (Dept. of Nat. Health and Welfare, Ottawa, Ont., Canada). *Clin. Chem.*, 1960, **6** (3), 243–253.—The method of Pearson *et al.* (*Anal. Chem.*, 1953, **25**, 813) is compared with six alternative methods and is found to be rapid, to yield comparable results, and to be suitable for routine purposes. The explosive hazard is not confirmed, but sulphosalicylic acid may be substituted for the toluene-p-sulphonic acid if desired. H. F. W. KIRKPATRICK

267. Paper chromatography of steroids and triterpenoids. II. Paper chromatography of sterols. Itsuo Nishioka (Pharm. Inst., Med. Fac., Kyushu Univ., Katakasu, Fukuoka). *J. Pharm. Soc. Japan*, 1959, **79** (11), 1453–1456.—Thirteen sterols

were separated by reversed-phase paper chromatography, with liquid paraffin as the stationary phase and a polar solvent system (*e.g.*, methanolwater, propanol - methanol - water or 2-ethoxyethanol - propanol - methanol - water) as the mobile phase. The relationship between structure and the R_F value of the sterols is discussed.

III. Paper chromatography of triterpenoids. Itsuo Nishioka. *Ibid.*, 1959, **79** (11), 1457–1461.— The paper chromatography of triterpenes and triterpenoid saponins was examined with a number of solvent systems on paper saturated with water, liquid paraffin or methyl stearate. The relationship between chemical structure and R_F value is discussed.   S. NATORI

268. Qualitative, quantitative and preparative chromatography of steroids on fully acetylated paper. II. Quantitative chromatography of △⁴- and △¹,⁴-3-oxosteroids. J. Hartel, A. Bol Raap and F. J. Ritter (Central Lab. T.N.O., Delft, The Netherlands). *J. Chromatography*, 1960, **3** (5), 482–487 (in English).—The method involves chromatography in benzene - methanol - water (4 : 4 : 1) on fully acetylated paper, the steroid spots being subsequently cut out, eluted and determined spectrophotometrically. Exact determination is possible because of the absence of impregnating agent and slight adsorption of the steroids on the paper. Results are presented for cholest-4-ene-3-one, progesterone, testosterone, cortisol and prednisolone.   S. M. MARSH

269. Glass-fibre-paper chromatography of adrenal and gonadal steroids. J. F. Dingman, M. C. Staub, E. Gaitan and G. Bazzano (Endocrine Unit, Univ. Sch. of Med., New Orleans, La., U.S.A.). *Clin. Chem.*, 1960, **6** (3), 228–232.—Glass-fibre paper No. X-934-A.H. (H. Reeve Angel & Co.) is cleaned by heating in an electric furnace at 1100° F for 30 min. Strips are soaked in 0.1 M KH_2PO_4, dried and stored in a desiccator ready for use. A miscible solvent system composed of various amounts of dimethylformamide (0 to 3% v/v) in benzene is suitable for most steroids. Chromatography is conducted at room temp., solvents are mixed immediately before use and poured into a covered glass jar of suitable size, the test soln. is spotted near the lower edge, and the strip is dried and lowered into the solvent, without preliminary equilibration, for ascending development. A strip 20 cm long is completely developed in approx. 15 min. The solvents are evaporated, the paper is sprayed with conc. H_2SO_4 and heated over a hot plate until the acid is volatilised. The charred steroids appear as grey - black spots on a white background. Average R_F values for a number of steroids are tabulated.   H. F. W. KIRKPATRICK

270. Double isotope derivative assay of aldosterone in biological extracts. B. Kliman and R. E. Peterson (Nat. Inst. of Arthritis and Metabol. Diseases, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1960, **235** (6), 1639–1648.—Dried extracts of plasma or urine are acetylated with [³H]acetic anhydride in the presence of pyridine to convert the aldosterone into the labelled diacetate. A measured amount of aldosterone [¹⁴C]diacetate is then added, and the doubly-labelled steroid is purified by paper chromatography. The sample is then treated with a 0·5% soln. of CrO_3 in acetic acid to form a mono-acetate oxidation product, and this is re-chromatographed to separate the product from other ³H-labelled

material. The contents of ³H and ¹⁴C are determined by counting in a liquid scintillation spectrometer. The amount of aldosterone present in the original extract is calculated from the determination of the amount of ¹⁴C indicator lost during purification, the yield of ³H radioactivity, and the specific activity of the [³H]acetic anhydride. The assay needs 5 to 30 ml of human urine or 2 to 3 ml of dog adrenal vein plasma. It is applicable to 0·01 µg of aldosterone, and has the accuracy and precision of radio-isotopic techniques.   J. N. ASHLEY

271. Method of determining aldolase and a means of expressing its activity. E. P. Korzunova (Smolensk Med. Inst.). *Lab. Delo*, 1960, **6** (3), 21–23.— Alterations are suggested to the method of Sibley and Lehninger (*J. Biol. Chem.*, 1949, **177**, 859). The soln. for incubation at 37° consists of barbitone buffer (pH 6·8) (1·5 ml), 0·05 M fructose 1,6-diphosphate (Na salt) (0·25 ml), 0·056 M hydrazine sulphate (0·25 ml), 0·002 M iodoacetic acid (0·25 ml), H_2O (0·05 ml) and test soln. (0·2 ml). The procedure is then as described by Sibley and Lehninger.   A. BURWOOD-SMITH

272. Notes on the hydroxamate assay for amino-acid-activating enzymes. I. D. Raacke and J. Bové (Virus Lab., Univ. of Calif., Berkeley, U.S.A.). *Experientia*, 1960, **16** (5), 195–196 (in English).— Sources of error in the hydroxamate assay are pointed out. The final pH (0·9) of the reaction mixture for the determination of hydroxamate after the addition of $FeCl_3$ is important. Over the pH range 0·9 to 0·3 there is a 20% decrease in colour yield per 0·1 pH unit. The apparent activity is also reduced to one-third when hydroxyammonium chloride soln. that has been stored for 21 days is used for the assay. In addition, the tissue extracts may destroy the amino-acid hydroxamates formed.   P. NICHOLLS

273. Rapid method for the quantitative determination of serum alkaline phosphatase. B. Klein, P. A. Read and A. L. Babson (Vet. Admin. Hosp., Bronx, N.Y., U.S.A.). *Clin. Chem.*, 1960, **6** (3), 269–275.—A commercial tablet form (Phosphatabs) of buffered phenolphthalein phosphate is used, the amount of phenolphthalein liberated under controlled conditions being determined photometrically to give a measure of the enzyme activity.   H. F. W. KIRKPATRICK

274. Colorimetric determination of serum isocitric dehydrogenase. T. H. Taylor and M. E. Friedman (Dr. Milton J. Goodfriend Res. Lab., Lebanon Hosp., New York, U.S.A.). *Clin. Chem.*, 1960, **6** (3), 208–215.—A method is described in which the α-oxoglutaric acid formed during the reaction is determined with 2,4-dinitrophenylhydrazine. The results are related directly to the spectrophotometric units of Wolfson and Williams-Ashman (*Proc. Soc. Exp. Biol., Med.*, 1957, **96**, 231).   H. F. W. KIRKPATRICK

275. Colorimetric assay of coenzyme I and II dependent enzymes. J. King (North Lonsdale Hospital, Barrow-in-Furness, England). *J. Med. Lab. Technol.*, 1960, **17** (2), 89–92.—Coenzyme I (diphosphopyridine nucleotide) and coenzyme II (triphosphopyridine nucleotide) form condensation products with 2,4-dinitrophenylhydrazine, and the effect of this on the colorimetric determination with 2,4-dinitrophenylhydrazine of enzymes requiring

these coenzymes is discussed. A modified standard curve for the determination of lactic dehydrogenase by the method of King (*Anal. Abstr.*, 1960, **7**, 3416) is suggested.   R. A. BRENAN

276. Quantitative determination of coenzyme A by sorbyl coenzyme-A formation. S. J. Wakil and G. Hübscher (Inst. for Enzyme Res., Univ. of Wisconsin, Madison, U.S.A.). *J. Biol. Chem.*, 1960, **235** (6), 1554–1558.—The spectrophotometric assay depends on the formation of sorbyl coenzyme A by interaction of coenzyme A with sorbic acid in the presence of the fatty acid-activating enzyme, adenosine triphosphate and Mg^{2+}, and measurement of the sorbyl coenzyme A at 300 mμ. The method is applicable to 1 to 15 mμmoles of coenzyme A, and can be used with tissue extracts.

J. N. ASHLEY

See also '*Abstracts*—**13**, Inorg. cations in biological fluids. **67**, Scintillation counting of ^{14}C. **170**, Detection of phosphate esters. **179**, I.r. spectra of fungal pigments. **333**, Determination of vitamin B_{12}. **352**, Fractionation of nucleic acids. **365**, Zaffaroni-type solvent system for paper chromatography. **370**, Use of bromophenol blue for spot tests. **385**, Tank for electrophoresis of proteins. **386**, Photometry of lipid electropherograms. **393**, Alkaline-earth metals in biological material.

Pharmaceutical analysis

277. Flame-photometric examination of pharmaceutical materials. E. Pungor and E. Király (Inst. f. anorg. u. anal. Chem., L. Eötvös Univ., Budapest, Hungary). *Arch. Pharm., Berlin*, 1960, **293** (5), 494–501.—The effects of acetic and ascorbic acids, H_3BO_3, $KMnO_4$ and Ca lactate in various concn. on the mol. and atomic emissions of Na^+ (and in some cases of Ca^{2+} and K^+) are examined. Emissions of NaCl are not affected by the presence of ascorbic acid. Suitable methods are given for the elimination of the various forms of interference caused by the other compounds mentioned. A suitable formula is given for dealing with the interference with the emissions of NaCl caused by the presence of KOH. The presence of >30% of ethanol causes undue interference with the emisssion of standard and test soln. The Zeiss flame-photometer gives satisfactory results with the direct use of an injector-atomiser, without a cloud-chamber atomiser.   P. S. ARUP

278. Radiochromatographic evaluation of alkaloids in tincture and extract of nux vomica. M. Šaršúnová, J. Tölgyessy and J. Majer (Inst. Anal. Chem., Pharm. Fac., Bratislava, Czechoslovakia). *Pharm. Acta Helv.*, 1960, **35** (6), 271–275 (in German).— Apply the tincture or the diluted extract to Whatman No. 1 paper, dry the spot in warm air and develop with water-saturated n-butanol by the descending technique for 16 hr. Immerse the chromatogram for 3 min. in a soln. of molybdophosphoric acid labelled with ^{32}P, remove the excess of reagent by washing with 2·5% HCl, allow to dry and scan with a Geiger - Müller counter. Determine the concn. of brucine and strychnine in the sample from the counts of radioactivity at the corresponding maxima. The recovery is about 95% with an error of $\simeq \pm 3\%$.   A. R. ROGERS

279. The use of paper chromatography in studying the stability of apomorphine. F. Machovičová, O. Mohelská and V. Parrák (State Inst. Control of Drugs, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1960, **9** (5), 243–246.—Apomorphine (I) can be separated from an oxidation product (II) formed in unstabilised aq. soln. by paper chromatography of the test soln. (containing 100 to 200 μg of I) with n-butanol - formic acid - water (77:13:10) (R_F values 0·5 and 0·7 for I and II, respectively) or n-butanol - acetic acid - water (4:1:5) (R_F values 0·65 and 0·85). Dragendorff's reagent gives an orange spot with I and a green one with II. For injection soln. stabilised with ascorbic acid or mannitol, the best spray reagent is bromophenol blue soln. containing borate, which gives yellow spots with ascorbic acid and mannitol, and a green one with I on a blue background.   J. ZÝKA

280. Colorimetric method for the determination of atropine by means of Reinecke's salt. S. Bartkowicz (Dept. of Appl. Pharm., Med. Acad., Cracow). *Dissert. Pharm.*, 1960, **12** (2), 113–120.—Ammonium reineckate (I) is used for the determination of atropine in 1% H_2SO_4. I soln. (0·5%) is added to the test soln., the mixture is placed in a refrigerator for 30 min., and the ppt. is collected on a glass filter, washed with cold water and dissolved in acetone. The extinction is then measured against a reagent blank. Alternatively, the extinction of the I soln. is measured before and after pptn. by the test soln. The difference is proportional to the amount of atropine reineckate pptd.   B. K.

281. Spectrophotometric determination of ephedrine hydrochloride. R. C. Shah, P. V. Raman and S. B. Gandhi (Mac Laboratories Private Ltd., Bombay). *Indian J. Pharm.*, 1960, **22** (6), 140–141.—The extinction at 207 mμ of a soln. in 0·1 N HCl is measured in a Beckman spectrophotometer, model G 2400, with DU photomultiplier combination G 4700, with a hydrogen discharge tube as a light source and 1-cm silica cells. Beer's law is obeyed in the range 5 to 20 μg per ml. Pseudoephedrine shows exactly the same u.v. absorption as ephedrine. The procedure has been successfully applied to the determination of ephedrine hydrochloride in tablets, elixir, ointment and nasal drops.   A. R. ROGERS

282. Colorimetric determination of dihydroxypropyltheophylline [diprophylline] by means of molybdophosphoric acid. T. Pelczar (Dept. of Inorg. and Anal. Chem., Med. Acad., Kraków). *Dissert. Pharm.*, 1960, **12** (2), 107–111.—Diprophylline (I) ($\simeq 10$ mg) is dissolved in water, conc. HCl is added, the soln. is heated and molybdophosphoric acid is added. The cooled ppt. is washed with dil. HCl and dissolved in 0·1 N NaOH. A soln. of ammonium molybdate in H_2SO_4 is added, followed by a soln. of ascorbic acid, then the mixture is boiled for 1 min., cooled, and measured colorimetrically. This method was used for the determination of I in tablets and injections.

B. K.

283. Reactions of deoxy-sugars. Application to the determination of glycosides. H. Wachsmuth and L. van Koeckhoven (Lab. de Chim. et de Biochim., Hôpital Stuivenberg, Anvers). *J. Pharm. Belg.*, 1959, **14** (3–4), 165–172.—Many reagents are studied for the characterisation and identification of deoxy-sugars, glycosides and similar substances. These include mineral salts, aldehydes, indole derivatives, heteropoly-acids, and the chlorides of

Sb, Bi and As. Colour reactions and test procedures are described. Quantitative methods have been proposed for two of the reagents, *viz.*, sodium molybdate and phthalaldehyde. In each case the extinction is compared with that of a standard sample. *Procedure with sodium molybdate*—Dissolve the deoxy-sugar or glycoside (25 to 200 μg) in 2 ml of acetic acid. Add 0·5 ml of molybdate soln. (0·5 ml of 50% aq. sodium molybdate in 25 ml of acetic acid), and then 0·5 ml of acetic acid containing 10% of H_2SO_4. Set aside for 3 hr., make up to 5 ml with acetic acid and measure the extinction at 675 mμ in a 1-cm cell for digitoxose (I), digitoxin (II) or digoxin. *Procedure with phthalaldehyde*: (a) *for* I *and* II—Dissolve the sample (25 to 200 μg) in acetone and evaporate to dryness. Add 0·1 ml of reagent (100 mg of phthalaldehyde in 4 ml of ethanol) and 6 ml of HCl. Set aside for 90 min. and read the extinction at 525 mμ for II, or 530 mμ for I. (b) *For* II—This procedure is used when method (a) gives a cloudy soln. Add 0·2 ml of reagent (0·2 g of phthalaldehyde in 4 ml of ethanol) and 6 ml of HCl to the sample (25 to 200 μg). Set aside for 1 hr., add 4 ml of butanol, set aside for 20 min., and read the extinction at 520 mμ. Method (b) may be slightly modified for application to deoxyribose.   R. A. HENDEY

284. Automated system for continuous determination of penicillin in fermentation media using hydroxylamine reagent. A. O. Niedermayer, F. M. Russo-Alesi, C. A. Lendzian and J. M. Kelly (Squibb Inst. for Med. Res., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 664–666.—The use of a Technicon AutoAnalyser is described, in which the complex obtained by the reaction of penicillin (I) with hydroxylamine and Fe^{3+} (*cf.* Boxer and Everett, *Brit. Abstr. C*, 1949, 404) is measured at 480 mμ. Beer's law is obeyed over the range 250 to 10,000 units of I per ml. The method, which is preferred to one based on differences in the iodine uptake of active and degraded I (*cf.* Ferrari *et al.*, *Anal. Abstr.*, 1960, **7**, 2431), has been used extensively for the assay of fermentation media. Results are in satisfactory agreement with those obtained microbiologically. The coeff. of variation is 2·5%.
     A. R. ROGERS

285. Determination of chloramphenicol in various medicaments. M. Andrey and A. Mirimanoff (Lab. de Pharm. Galén., Univ. de Genève, Switzerland). *Pharm. Acta Helv.*, 1960, **35** (6), 276–283 (in French).—Extract suppositories with benzene and use the benzene-insoluble portion; extract the contents of capsules with absolute ethanol and evaporate to dryness. Ignite the dried residue with Na_2CO_3, extract with H_2O and determine Cl⁻ by the method of Volhard. The precision of the method is within $\simeq \pm 6\%$.   A. R. ROGERS

286. Rifomycin. VII. Spectrophotometric analysis of rifomycin B. G. G. Gallo, P. Sensi and P. Radaelli (Lab. Ric., Lepetit S.P.A., Milano, Italy). *Farmaco, Ed. Prat.*, 1960, **15** (5), 283–291.—Spectrophotometry is applicable to the determination of rifomycin B as pure substance, and in pharmaceutical preparations, fermentation broths and biological fluids. In pure solution in phosphate buffer at pH 7·3 (2·28 g of $NaH_2PO_4.H_2O$ and 18·4 g of $Na_2HPO_4.12H_2O$ per litre), $E_{1\,cm}^{1\%}$ at 425 mμ = 220. Pharmaceutical preparations are dispersed in the buffer and the rifomycin is extracted into ethyl acetate after acidification to pH 2. For rifomycin B in ethyl acetate, $E_{1\,cm}^{1\%}$ at 414 mμ = 145.

If the ethyl acetate extract is coloured (*e.g.*, in the assay of fermentation broths and biological fluids) the rifomycin is re-extracted into pH 7·3 buffer for spectrophotometric determination. The absorption spectra of rifomycin B in various solvents and at different pH values are reproduced over the range from 250 to 500 mμ.

VIII. Fluorimetric assay of rifomycin B. P. Sensi, G. Coronelli and A. Binaghi. *Ibid.*, 1960, **15** (5), 292–300.—The method is based on measurement of the fluorescence of the triacetyl derivative in ethyl acetate soln., which has an emission maximum at 500 mμ. Solid samples are dissolved in 0·067 M phosphate buffer at pH 7·3 (approx. 20 μg of rifomycin B per ml). The soln. is acidified to pH 2 with 10% HCl and extracted twice with an equal volume of ethyl acetate. The extract is dried with anhyd. Na_2SO_4, filtered and made up to a known vol. An aliquot containing from 50 to 500 μg of rifomycin is measured into a 50-ml flask, acetic anhydride (0·1 ml) and pyridine (0·2 ml) are added and the mixture is diluted to vol. with ethyl acetate and set aside for 2 hr. The fluorescence at 500 mμ is then measured in a spectrofluorimeter and compared with the fluorescence of standards treated similarly. The graph of fluorescence *vs.* concn. is linear over the range from 1 to 10 μg per ml. The standard deviation at the 10-μg level is 0·42 μg per ml.   E. C. APLING

287. Methods of insulin assay. G. A. Stewart (Biological Control Laboratories, The Wellcome Foundation, London). *Brit. Med. Bull.*, 1960, **16** (3), 196–201.—A review, with 83 references.
     D. E. EVANS

288. Determination of some alkaline organic salts in non-aqueous medium. R. Vasiliev, E. Şişman, M. Jecu and I. Chialda (Inst. Pharm. Res., Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (6), 347.—To approx. 1·5 to 2 milli-equiv. of organic salt dissolved in 20 ml of ethanol is added $\simeq 2$ g of phenol and the mixture is titrated with 0·1 N $HClO_4$ in dioxan to thymol blue. Errors of $\simeq \pm 0·13\%$ are shown for a number of salts.
     H. SHER

289. Paper chromatography of derivatives of barbituric acid. A. Macek (Forschungsinst. f. Pharm. u. Biochem., Prague, Czechoslovakia). *Arch. Pharm., Berlin*, 1960, **293** (5), 545–550.—R_F values obtained with the use of benzene, $CHCl_3$, or benzene - $CHCl_3$ (1:1) as descending solvents on paper impregnated with formamide are given for 41 substitution derivatives of the acid. The values for di- and tri-substitution derivatives are spread over wider ranges than those obtained by the usual techniques. Barbituric acid and its mono-substitution derivatives remain at the starting point, and can subsequently be treated with other solvents. Constants are given for 20 possible substituting groups, showing their influence on the R_F value (obtained with $CHCl_3$ as solvent) when replacing an ethyl group in the C_5 position.   P. S. ARUP

290. The application of alternating-current polarography in the determination of phenobarbitone in dosage forms. N. G. Lordi, E. M. Cohen and B. L. Taylor (College of Pharmacy, Rutgers, The State Univ., Newark, N.J.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 371–375.—Prepare a soln. containing phenobarbitone (0·2 to 0·4 mg) in a M soln. of KNO_3 in borate buffer of pH 7·9 (25 ml) and submit it to polarography with an applied alternating current of 0·03 V r.m.s. and a rate of

scan of 0·00062 V per sec. Calculate the concn. of phenobarbitone from the peak current by reference to a standard curve. The accuracy is $\simeq \pm 2\%$. Caffeine, diphenylhydantoin, aminophylline, theobromine sodium salicylate, thiamine, polyoxyethylene glycol 400 and halides interfere.

A. R. ROGERS

291. Determination of amidopyrine in non-aqueous medium. R. Vasiliev, V. Scintee and I. Chialda (Inst. Pharm. Res., Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (6), 347.—*Procedure*—Dissolve between 0·3 and 0·4 g in 20 ml of $CHCl_3$ and titrate with 0·1 N $HClO_4$ in dioxan until methyl red changes to red - violet. The error is $\pm 0.04\%$. It is claimed that the method can be used for mixtures containing salts of alkaloids, caffeine and other purine and barbituric acid derivatives.

H. SHER

292. Colorimetric determination of 4-aminophenazone and 4-formamidophenazone in amidopyrine. J. Gasparič (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Ceskosl. Farm.*, 1960, **9** (5), 241–243.—*Procedure*—Dissolve amidopyrine (4 g) in 0·4 N HCl (50 ml). Cool an aliquot (10 ml) to 0° to 5° and add $NaNO_2$ soln. (2%) (2 ml). After 5 min. add a soln. of phenyl-J-acid (dissolve 6 g of anhyd. Na_2CO_3 in 80 ml of H_2O, cool with ice, add 100 mg of phenyl-J-acid and dilute with H_2O to 100 ml; the soln. must be freshly prepared and stored at 0° to 5°) (10 ml), dilute with H_2O to 50 ml and after 10 to 15 min. measure the extinction at 490 mμ for 4-aminophenazone. Heat another aliquot for 30 min. on a boiling-water bath to hydrolyse 4-formamidophenazone to 4-aminophenazone, cool and proceed as described above to determine the sum of the two impurities. Beer's law is obeyed over the range 0 to 200 μg of 4-aminophenazone in 50 ml. The max. error is $\pm 5\%$.   J. ZÝKA

293. Determination of some tranquillisers by ammonia distillation. P. M. Parikh, G. T. Rajkotia and S. P. Mukherji (Zandu Pharmaceutical Works Ltd., Bombay). *Indian J. Pharm.*, 1960, **22** (6), 148–149.—Heat the sample (250 mg of meprobamate or 400 mg of mephenesin carbamate or methocarbamol) under reflux with 50% (v/v) H_2SO_4 soln. (10 ml) for 10 min., dilute with H_2O (40 ml), add 50% (w/v) NaOH soln. (25 ml), distil the liberated NH_3 into 0·1 N H_2SO_4 (50 ml) and titrate the excess of acid with 0·1 N NaOH, with methyl red as indicator. Perform a blank determination. The results are comparable with those obtained by the Kjeldahl method and are produced more quickly.   A. R. ROGERS

294. Photometric determination of Novazin (2-diethylaminoethyl phenothiazine-10-carboxylate). M. Sterescu, M. Popa and H. Aftalion. *Rev. Chim., Bucharest*, 1960, **11** (6), 346–347.—The method is based on pptn. with ammonium reineckate. The ppt. is dissolved in acetone and the extinction is measured at 530 mμ with water as blank. The error is $\pm 3\%$.   H. SHER

295. pH-Induced shifts in ultra-violet maxima for determination of isoniazid and acetylisoniazid in mixtures. Q. C. Belles and M. L. Littleman (Leahi Hosp., Honolulu, Hawaii, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 720–721.—Acetylisoniazid (**I**) and isoniazid (**II**) exhibit maxima at 265 mμ in acid soln. and at $\simeq 300$ mμ in alkaline soln. The

compounds make this shift to 300 mμ at different pH values, and can thus be differentiated; **I** makes a complete shift in max. at pH 10 while **II** remains unaffected; the complete shift for **II** occurs at pH 12. These u.v. differences were used to determine the compounds in blood, serum or urine. The method is useful when the sensitivity required is $\not< 1$ μg per ml.   G. P. COOK

296. Photometric determination of hydrochlorothiazide and its hydrolysis product. C. R. Rehm and J. B. Smith (Ciba Pharmaceutical Products Inc., Summit, N.J.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 386–389.—The u.v. absorption max. at 271 mμ in methanol or H_2O may be used for the quant. determination of hydrochlorothiazide (**I**) in the absence of the main hydrolysis product 4-amino-6-chlorobenzene-1,3-disulphonamide (**II**); Beer's law is obeyed over the range 1 to 10 mg per litre. To determine **II** in the presence of **I**, dissolve the sample in methanol or 2-methoxyethanol and dilute with H_2O; mix the soln. (2 to 5 ml containing $\simeq 0.1$ mg of **II**) with 1% $NaNO_2$ soln. (1 ml) and N HCl (5 ml), set aside for 3 to 5 min., add 2% ammonium sulphamate soln. (1 ml), mix, set aside for 3 min., add a fresh 0·5% soln. of chromotropic acid (disodium salt) (1 ml) and N Na acetate (10 ml), dilute to 50 ml with H_2O, mix, and measure the extinction at 500 mμ against a reagent blank. Treat similarly a standard soln. of **II**. To determine **I** and **II** together, mix a soln. of the sample ($\simeq 0.2$ mg) in 0·5 N aq. NH_3 (2 ml) with 0·7% hydroxyammonium chloride soln. (0·5 ml), heat at 100° for 60 min. in a closed flask, then cool, and determine **II** as described above. Calculate the amount of **I** by difference. The precision is $\simeq \pm 1\%$.

A. R. ROGERS

297. Identification and spectrophotometric determination of hydrochlorothiazide (Hydrodiuril). M. Sterescu, V. Topa, S. Arizan, S. Iacobescu and Z. Teodorescu (Inst. Phys., Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (5), 296–297.—The proposed method is based on the characteristics of the absorption curves of a soln. of hydrochlorothiazide (**I**) in 0·0002 N NaOH, *viz.*, max. extinctions E_1 at 222 mμ and E_2 at 274 mμ and the relationship $1.9 < E_1/E_2 \not> 2$. *Procedure*—The sample (0·25 g) is dissolved in 50 ml of 0·1 N NaOH; 2 ml is then diluted to 100 ml with water and 5 ml of this soln. is then further diluted with 50 ml of water (at concn. $>$ 10 μg per ml, **I** is not completely soluble). The pure compound gives values for $E_{1cm}^{1\%}$ of $E_1 = 0.990$ and $E_2 = 0.495$. The concn. of a sample in μg per ml is then given by $10/0.99$ E_1 or $10/0.495$ E_2. The results obtained with a Zeiss spectrophotometer gave errors of $<2\%$.   H. SHER

298. New method for the determination of 4-amino-6-chlorobenzene-1,3-disulphonamide. S. Arizan and M. Sterescu. *Rev. Chim., Bucharest*, 1960, **11** (5), 297–298.—The method for the determination of 4-amino-6-chlorobenzene-1,3-disulphonamide (**I**) as an impurity in hydrochlorothiazide is based on the colour reaction given by **I** when diazotised and coupled with N-1-naphthylethylenediamine (**II**). *Preparation of a standard curve*—**I** (0·025 g) is dissolved in 5 ml of N NaOH and diluted to 250 ml, from which aliquots of 0·1, 0·2 and 0·3 ml are taken. Each is diluted to 2 ml with 0·02 N NaOH, stirred and treated with 2 ml of 0·1 N HCl and 0·4 ml of a 1% soln. of $NaNO_2$, stirred and set aside for 5 min. Each is treated with

0·4 ml of a 0·4% sulphamic acid soln., stirred for 1 min., and 0·4 ml of a 0·1% aq. soln. of the di-hydrochloride of **II** is added, with stirring. The extinctions of the rose-coloured soln. are read after 10 min. at 530 mμ in a 1-cm cell against water. *Determination of* **I** *in chlorothiazide*—The sample (0·2 g) is dissolved in 2 ml of N NaOH and diluted to 100 ml. The soln. (1 ml) is treated with 1 ml of 0·02 N NaOH and the procedure for the standard curve is followed. The error is ≯ ±4%.   H. SHER

299. Microchemical reactions for some new oral diuretics. H. Groenewegen (Apoth. Ziekenf. "De Volharding," Den Haag, Netherlands). *Pharm. Weekbl.*, 1960, **95** (11), 345–350.—Microchemical reactions are described for chlorothiazide (**I**), hydrochlorothiazide (**II**), hydroflumethiazide (**III**) and acetazolamide (**IV**). The following reagents are used—aq. NH₃ (for **I**, **III** and **IV**), Cu acetate and aq. NH₃ (for **I**), Zn acetate and aq. NH₃ (for **I**, **III** and **IV**), AgNO₃ and aq. NH₃ (for **I**), ethanolamine and AgNO₃ (for **I** and **IV**), ethylenediamine and AgNO₃ (for **I**, **III** and **IV**) and the reagents of Bouchardat and Dragendorff - Amelink (for **I** and **II**). Twenty illustrations showing the crystal form of the products are given.   M. J. MAURICE

300. Volumetric determination of nitrofurazone. P. Spacu and G. Teodorescu (Univ. "C.I. Parhon," Bucharest, Romania). *Z. anal. Chem.*, 1960, **174** (5), 321–322 (in German).—Nitrofurazone (5-nitro-2-furfuraldehyde semicarbazone) (**I**) is determined by hydrolysing to liberate an equivalent of hydrazine, which is titrated with standard chloramine-T soln. *Procedure*—To the sample soln. containing 0·03 to 0·07 g of **I** in dil. acetic acid (20 to 50 ml) add conc. HCl (20 to 30 ml) and water (20 to 30 ml). Heat to 70° to 80°, add methyl red - methylene blue indicator, and titrate with 0·1 N chloramine T, adding more indicator just before the end-point; 1 ml of 0·1 N chloramine T ≡ 0·004953 g of **I**. The maximum error for the given range is −0·4%.   W. T. CARTER

301. Quantitative colorimetric determination of residual 9α-fluoroprednisolone and 9α-fluorohydrocortisone in triamcinolone samples. C. J. Sih, S. C. Pan and R. E. Bennett (Squibb Inst. for Med. Res., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 669–671.—Dissolve the sample, containing 10 to 100 μg of 9α-fluoroprednisolone (**I**) or 9α-fluorohydrocortisone (**II**) or both, in methanol (0·1 ml), cool in ice and add successively H₂O (0·4 ml), 0·01% fructose soln. (0·5 ml) and H₂SO₄ (mix 6 vol. of conc. H₂SO₄ with 1 vol. of H₂O) (4·5 ml). Cool in ice for 10 min., with occasional shaking, set aside at room temp. for ≃ 10 min. and then heat at 60° for exactly 5 min. Cool in water, add 3% L-cysteine hydrochloride soln. (0·1 ml) and set aside overnight at room temp. Dilute with H₂O (1·2 ml) and measure the extinction at 548 mμ and at 625 mμ. Simultaneously run a blank containing pure triamcinolone (**III**). The absorption at 625 mμ is due to **I** and that at 548 mμ is due to **I** plus **II**. When the amount of **III** present is > 50 times the amount of **I** plus **II**, the chromogens do not obey Beer's law, and a standard curve must be prepared. The precision is ≃ ±3%. Many other steroids give colour reactions with the reagents.   A. R. ROGERS

302. Analysis of anaesthetic mixtures by gas chromatography. E. R. Adlard and D. W. Hill ("Shell" Research Ltd., Thornton Research Centre,

Chester, England). *Nature*, 1960, **186**, 1045.—To separate O and N, use a column (1 ft. × 0·25 in.) of 52 to 60-mesh Linde 5A molecular sieve at room temp. To separate CO₂, N₂O and O, use a column (20 ft. × 0·25 in.) containing 20% of dimethyl sulphoxide on 52 to 60-mesh "SIL-O-CEL" at 20°. When ethyl ether and fluothane are also present, use a column (2 ft. × 0·25 in.) containing 15% of dinonyl phthalate on firebrick at 75° in parallel with the dimethyl sulphoxide column. With H as carrier gas at a flow rate of 30 ml per min., analysis is complete in 4·5 min.   A. R. ROGERS

303. Application of paper electrophoresis to the detection of heavy metals as impurities in pharmaceutical preparations. D. Małysz (Drug Res. Inst., Warsaw). *Acta Polon. Pharm.*, 1960, **17** (3), 221–228.—Conditions for the use of electrophoresis are investigated. The best results are given by the following procedure. Incinerate 1 g of sample, add a few drops of conc. HCl, dry, dissolve the ash in 1 ml of 0·1 N HCl, with heating, and filter through a small filter. Transfer 0·05 to 0·2 ml of the filtrate to Whatman No. 1 paper and apply a potential of 300 V for 3 hr.; use Na citrate buffer (10 g of citric acid and 3·5 g of NaOH in 1 litre of water). The following reagents were used for detection (figures in parentheses show the smallest detectable amount)—*p*-phenetidine for Fe (0·5 μg), Cu (0·25 μg) and Zn (0·25 μg); 8-hydroxyquinoline for Fe (0·25 μg), Cu (0·5 μg) and Zn (0·25 μg); Na diethyldithiocarbamate for Fe (0·25 μg) and Cu (0·1 μg); rhodizonic acid for Pb (0·2 μg); and dithizone for Pb (0·25 μg) and Zn (0·1 μg). A modification is described for preparations containing Na and Ca in which the heavy metals are pptd. with H₂S in the presence of mercury salts.   B. K.

304. Complexometric method for the assay of strong solution of lead subacetate B.P. C. R. Mehta, N. V. Mankad and M. B. Devani (Dept. of Pharm. Chem., L. M. Coll. of Pharmacy, Ahmedabad, India). *Indian J. Pharm.*, 1960, **22** (5), 123–124.—*Procedure*—Dilute the sample (1 g) with cation-free H₂O (20 ml) and titrate with 0·1 M EDTA (disodium salt), with continuous shaking, until the turbidity changes to a ppt. with a clear supernatant liquid (*a* ml). Continue the titration with vigorous shaking until the ppt. dissolves and a clear soln. is obtained (*b* ml). The results for total lead as Pb (calculated from *b*) are 0·44% (absolute) higher than by the B.P. method, but agree closely with those by a gravimetric sulphate method. The results for alkalinity as PbO, calculated from (*b* − *a*), are also 0·44% higher than by the B.P. method. The standard deviations, based on 10 replicates, were 0·024% for the proposed method and 0·033% for the B.P. method.   A. R. ROGERS

305. Analysis by gas chromatography of lozenges "a l'éther." L. Maricq and L. Molle (Univ. Libre de Bruxelles). *J. Pharm. Belg.*, 1959, **14** (3–4), 156–158.—The chloroform, ethanol and ethyl ether contents are determined directly. *Procedure*—Introduce a lozenge into a stoppered flask containing 10 ml of H₂O. Set the flask aside for 1 to 2 hr., shaking at intervals until the lozenge has disintegrated. Centrifuge for 10 min. in a stoppered tube at 2500 to 3000 r.p.m. Inject 10 μl of the supernatant liquid into the gas stream of the chromatograph. The column (4 metres) is packed with 20% of Carbowax 1500 on Celite. The operating temp.

is 106° ± 0·5° and the carrier gas is H, at a flow rate of 25 ml per min.   The content of $CHCl_3$ is obtained by comparing the peaks with a standard curve obtained in the same way; the amounts of ethanol and ether can also be measured.   Results compare well with those found by a preliminary distillation followed by gas chromatography.

R. A. HENDEY

306. Estimation of the biological potency of iso-merised vitamin-A palmitate, in aqueous multi-vitamin dispersions, from maleic values. S. R. Ames, W. J. Swanson and R. W. Lehman (Distillation Products Industries, Div. of Eastman Kodak Co., Rochester, N.Y.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 366–368.—The relative biopotency (y) is the potency determined by the slope-ratio liver-storage method (*cf.* Ames and Harris, *Anal. Abstr.*, 1956, **3**, 3202) expressed as a percentage of the chemical potency determined by the blue colour with $SbCl_3$.   The maleic value (x) is determined by the method described by Embree *et al.* ("Methods of Biochemical Analysis," Vol. 4, Interscience Publ. Inc., New York, 1957, p. 43).   Studies of stored multivitamin dispersions showed that y and x are related by the cubic regression equation—

$$y = 99 \cdot 5 - 0 \cdot 2x - 0 \cdot 051x^2 + 0 \cdot 000768x^3.$$

The isomerisation of all-*trans* vitamin-A palmitate in aq. dispersions appears to reach an equilibrium; y does not fall below 60% even after prolonged storage.   A. R. ROGERS

307. Determination of nordihydroguaiaretic acid antioxidant in oily vitamin preparations. A. Engelund (Denmark Apothekerforenings Kontrollab., Copenhagen). *Pharm. Acta Helv.*, 1960, **35** (6), 284–291 (in English).—Extract nordihydro-guaiaretic acid (**I**) from fatty material with hot 63% ethanol, cool and filter the extract, and apply an aliquot (3 to 6 μl containing $\simeq 2\ \mu g$ of **I**) to S. & S. paper No. 2043B. Develop with a 2·5% aq. soln. of ethyl acetoacetate by the descending technique for 2 hr., dry the chromatogram, spray with ethanolic $AgNO_3$ soln., dry again and spray with aq. NH_3. Measure the area of the light-brown spot at $R_F \simeq 0\cdot 3$; the area is directly proportional to the log of the amount of **I**. The error is $\simeq \pm 15\%$.

A. R. ROGERS

See also Abstracts—**153**, Mercury in org. compounds. **157**, Ethanol in aq. media. **176**, Polycyclic hydrocarbons in tobacco smoke. **185**, I.r. spectra of naphthols. **200**, Ethanol in volatile oils. **236**, Identification of corticosterone sulphate. **268**, Chromatography of medicinal steroids. **331**, Determination of thiamine. **333**, Determination of vitamin B_{12}. **342**, Determination of warfarin.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

308. Studies on legume starches. IV. Separation and determination of amylose and amylopectin. (2). Iodine coloration and potentiometric titration. Sin'itiro Kawamura and Minoru Tada (Fac. of Agric., Kagawa Univ., Miki-cho, Kagawa-ken). *J. Agric. Chem. Soc. Japan*, 1959, **33** (4), 296–300.— Amylose and amylopectin were separated from eight kinds of legume starch and potato starch by dispersion in 1% NaOH soln. and pptn. with butanol. The amounts of starch, amylose and amylopectin were determined by colorimetry (McCready *et al.*, *Anal. Chem.*, 1950, **22**, 1156) and potentiometric titration (Bates *et al.*, *J. Amer. Chem. Soc.*, 1943, **65**, 142).   S. NATORI

309. Analytical applications of the complexans. XXVII. Determination of heavy metals in samples of cane and beet syrup with ADCT [1,2-diaminocyclo-hexane-*NNN'N'*-tetra-acetic acid] and AEGT [1,2-di-(2-aminoethoxy)ethane-*NNN'N'*-tetra-acetic acid]. F. Bermejo Martínez, M. Paz Castro and R. Rey Mendoza (Fac. de Ciencas, Santiago de Compostela, Spain). *Inf. Quim. Anal.*, 1960, **14** (3), 61–63, 80.—The syrup (0·5 g) is oxidised with HNO_3 and H_2SO_4 and evaporated to dryness, and the residue is dissolved in dil. HCl and diluted to 250 ml. To aliquots (25 ml) are added 0·01 M ADCT (disodium salt) (5 ml) and 0·01 M AEGT (disodium salt) (5 ml); the pH is adjusted to 11 to 12 by addition of aq. NH_3 and murexide is added as indicator. The excess of complexan is determined by back-titration with 0·01 M Ca^{2+}. The ADCT titration gives the sum of Al, Fe, Cu and Zn, and the AEGT titration gives the sum of Cu and Zn; Al and Fe are obtained by difference.   G. H. FOXLEY

310. Examination of the occurrence of honeydew in honey. K. C. Kirkwood, T. J. Mitchell and D. Smith (Dept. of Chem. Technol., Royal Coll. of Science and Technology, Glasgow, Gt. Britain). *Analyst*, 1960, **85**, 412–416.—The insect excretion, honeydew, collected by bees during shortage of floral nectar, affects the quality of the honey and the health of the bees. It has a dark colour and rank flavour, and can be distinguished from floral honey by its high content of ash, dextrin, sucrose and reducing sugars, its higher pH and free acidity and its dextro-rotation. The relative values of these properties for detecting honeydew in honey were examined by methods briefly described, but, owing to overlapping of the ranges, these results proved of little value for distinguishing floral honey from honeydew honey with certainty. They were therefore subjected to statistical discriminatory analysis, and mathematical investigation showed that an efficient discriminant function is given by the equation $X = -\ 8 \cdot 3x_1 - 12 \cdot 3x_2 + 1 \cdot 4x_3$, in which x_1 is the pH, x_2 the ash (%) and x_3 the reducing sugars (%). In practice, a honey having a value of $X > 73 \cdot 1$ may be classed as floral and one having a value of $X < 73 \cdot 1$ as a honeydew honey. With samples having a value of X near 73·1 the dark colour, unpleasant flavour, dextro-rotation, high dextrin content and high acidity provide confirmatory evidence of the presence of honeydew.

A. O. JONES

311. Correlation of anticholinesterase effect with solanine content of potatoes. D. C. Abbott, K. Field and E. I. Johnson (D.S.I.R., Lab. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1960, **85**, 375–376.—Results obtained for the solanine content of new potatoes determined by the spectrophotometric method of Baker *et al.* (*Anal. Abstr.*, 1955, **2**, 3481) differed from those determined as the anticholinesterase activity of the juice expressed as the equivalent solanine content. Investigation showed that the anticholinesterase activity appears to have an approx. direct relation to the solanine content as determined by the spectrophotometric method. The methods used

are not specific for solanine, and the results cannot prove or disprove the implied opinion of Pokrovsky (*Biokhimiya*, 1956, **21**, 683) that the anticholinesterase activity is due principally to solanine, but they indicate, contrary to the findings of Orgall *et al.* (*Science*, 1958, **128**, 1136), that over 90% of the total anti-esterase is pptd. at pH 9·4.

A. O. JONES

312. Rapid determination of the protein content of milk. I. Colorimetric method with Amido black 10B and Orange G. K. Shiga, R. Shimizu and H. Hamada. *Bull. Nat. Inst. Agric. Sci., Japan, Ser. G*, 1959, (18), 1–6.—Milk (10 ml), diluted 1 in 20, was mixed with 25 ml of 0·001 M Amido black 10B (C.I. Acid Black 1), pH 2·8, and filtered after 5 min. The filtrate was diluted to 50 vol. and the extinction was measured spectrophotometrically at 610 mμ. This value, minus that of the blank, is a measure of the amount of dye adsorbed by the protein and is related to it by the equation $y = 0.063x + 0.013$, where x is the protein determined by the Kjeldahl method. Deviation from the results obtained by the Kjeldahl method on milk with a protein content of 1·5 to 4·5% was 0·083%. Amido Black 10B was superior to Orange G.

NUTR. ABSTR. REV.

313. Determination of sugars in fruits. II. Micro-determination of glucose in fruits by the hypoiodite method. Kazuo Osodo, Hidejiro Kazumi, Masayuki Kotaka and Hanao Shitomi (Nat. Inst. for Rural Ind., Shinjo, Yamagata, Japan). *J. Agric. Chem. Soc. Japan*, 1959, **33** (3), 166–170.—The conditions for the determination of glucose in the presence of fructose and sucrose by hypoiodite were examined.

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314. Identification of stored-products insects by the micromorphology of the exoskeleton. X. Common fragments of mill and grain moths. M. Harris (Dept. of Health, Education and Welfare, Food & Drug Admin., New Orleans, La., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1960, **43** (2), 444–458.—Characteristics of the wings, labial palpi, legs and antennae of various flour, meal and grain moths are described and illustrated.

A. A. ELDRIDGE

315. Paper-chromatographic and polarographic determination of vanillin and ethylvanillin in foodstuffs. II. H. Woggon, K. Rauscher and U. Köhler (Inst. f. Ernährung, Potsdam-Rehbrücke, Berlin). *Nahrung*, 1960, **4** (4), 374–388.—*Procedure* —The sample (5 g) is treated with abs. ethanol (10 ml) for 30 min. at 45°. The cooled extract is decanted off and a suitable aliquot (*e.g.*, 0·1 ml) is applied to Whatman 3MM paper with a micrometer pipette (illustrated). The paper is dried and developed by the ascending technique for 15 to 16 hr. with isobutyl alcohol - conc. aq. NH$_3$ (5:3); R_F values for vanillin are 0·33 to 0·40, and for ethylvanillin 0·51 to 0·58. The spots are located under u.v. light and immediately eluted with 0·5 M semicarbazide hydrochloride in 0·1 N HCl, and the eluates are polarographed between -0.5 and -1.0 V *vs.* the N.C.E. For vanillin (5 to 15 μg per ml) the mean error is $\pm 2\%$, and for ethylvanillin (2 to 5 μg per ml) it is $\pm 4\%$; concn. < 2 μg per ml can be determined with a mean error of $\pm 7\%$. Results are quoted for various flavoured sugars and pudding mixtures.

R. E. E.

316. Identification of carob-bean flour in dry gravy and mayonnaise mixtures. A. T. Czaja (Botan. Inst. der Tech. Hochschule, Aachen). *Z. LebensmittUntersuch.*, 1960, **112** (3), 190–194.—This flour, used in the textile industry for dressings and as a tragacanth substitute in the food and pharmaceutical industry, cannot be identified by chemical means and, as the husk and the embryo are removed during manufacture, the product loses morphological characteristics important for microscopical identification. But if the cell walls are cut lengthwise the tubular cells light up strongly under polarised light, and the cross-section shows round and oval cells which light up with varying intensity.

I. DICKINSON

317. Analytical studies of dyes by the use of infra-red spectra. I. Studies on yellow dyes for food. Michio Suzuki, Eizo Nakamura and Yuzo Nagase (Coll. of Pharm., Kashiwagi, Shinjuku-ku, Tokyo). *J. Pharm. Soc. Japan*, 1959, **79** (9), 1116–1119.—Infra-red spectroscopic analysis of the yellow dyes Naphthol yellow S (C.I. Acid Yellow 1), Yellow AB (C.I. Solvent Yellow 5), Yellow OB (C.I. Solvent Yellow 6), tartrazine, Sunset yellow FCF (C.I. Food Yellow 3), auramine and Butter Yellow was examined by the use of KBr discs, and the dyes were detected down to $\simeq 40$ μg by their key bands at 796, 992, 1109, 1008, 903, 945 and 767 and 928 cm^{-1}, respectively.

II. Studies on green dyes for food and malachite green. Michio Suzuki, Eizo Nakamura and Yuzo Nagase. *Ibid.*, 1959, **79** (9), 1209–1211.—Guinea green B (C.I. Acid Green 1) (**I**), Light green SF (C.I. Acid Green 5) (**II**), Fast green FCF (**III**) and malachite green (**IV**) (up to 40 μg each) were similarly analysed, the absorptions at 938 cm^{-1} for **I**, 1012 for **II**, 893 for **III**, 1042 for **I**, **II** and **III**, and 945 for **IV** being used as key bands.

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318. Analytical study of the natural red food dye alkannin. G. Dušinský and A. Szokolay (Inst. f. Hygiene, Bratislava, Czechoslovakia). *Nahrung*, 1960, **4** (4), 356–362.—Alkannin is present in the roots of *Alkanna tinctoria* L.; it is sol. in fats and org. solvents, has a melting range of 143° to 149° and is a permitted food dye in several countries. The absorption spectra (visual and u.v.), polarographic reduction and paper and adsorption chromatography of alkannin were studied. Procedures and results are given. Extinctions at 490 and 380 mμ are suitable as a criterion of purity, polarography is suitable for quant. determination, and paper chromatography or circular chromatography on a surface of Al$_2$O$_3$ is suitable for identification in the presence of other dyes.

I. DICKINSON

319. Rapid spectrophotometric method for simultaneous determination of benzoic and sorbic acids in margarine and butter. J. B. Roos and A. Versnel (Reichsuntersuchungsanst. für Molkereiprodukte und Nahrungsfette, Leiden, Netherlands). *Dtsch. LebensmittRdsch.*, 1960, **56** (5), 128–133.—A 5-g sample is warmed slightly and washed into a 100-ml flask with 30 ml of specially purified methanol; 10 ml of H$_2$O and 0·5 ml of 4 N H$_2$SO$_4$ are added, the mixture is warmed on a water bath to melt the fat, then shaken vigorously for 1 min., cooled to 20° and made up to vol. with methanol. The flask is placed in ice-water to solidify the fat and the soln. is filtered through a folded 15-cm S. & S. 588 paper, the first 20 ml being discarded. The extinctions at 220, 228, 233·3, 236, 252, 258, 264, 267, 272

and 277 mµ are measured in a quartz cell of thickness giving values between 0·2 and 0·8, and are calculated to a standard 1-cm thickness. For the comparison soln., 11 ml of H_2O and 0·5 ml of 4 N H_2SO_4 are made up to 100 ml with methanol. Calculation of the results is based on maxima at 228 mµ and 272 mµ for benzoic acid, 258 mµ for sorbic acid, and an isosbestic point at 233·3 mµ for the mixed acids. Readings taken on either side of the critical wavelengths allow corrections to be made for non-specific general absorptions. Small residual corrections are required for traces of fat extracted by the solvent. Recoveries within ≃ 50 p.p.m. are reported.　　　　C. L. HINTON

320. Presumptive detection of preservatives in canned cured-meat products by a simple yeast-growth test. D. A. A. Mossel, H. Zwart and A. S. de Bruin (Central Inst. for Nutrition and Food Res., T.N.O., Utrecht, The Netherlands). *Analyst*, 1960, **85**, 410–412.—The sample is homogenised at 60° with a substrate containing glucose, meat extract, tryptone and peptonised milk agar, and the pH is adjusted to 7·0 ± 0·2 with sterile NaOH soln. A similar portion of the sample is homogenised similarly, but the pH is adjusted to 3·6 ± 0·1 with tartaric acid soln. The mixtures, dried in Petri dishes at ≃ 35°, are inoculated with two V-shaped streaks of an emulsion in sterile saline of a subculture of *Candida brumptii* (prep. described) and are incubated at 30° ± 2° for 24 hr., the growth being observed visually. A blank test is made with a sample of the same kind of meat product known to be free from preservatives, the concn. of curing salts being adjusted to about the same level as in the test sample. The level of detection is the concn. of antimicrobial agent in the meat product that gives no growth or only a few punctiform colonies, copious development being observed in the blank. The levels of detection of preservatives in ham are quoted.　　　　A. O. JONES

321. Biphenyl and o-phenylphenol as preservatives for citrus fruit: a simple method for detection and determination of o-phenylphenol. M. Ihloff and M. Kalitzki (Chem. u. Lebensmittel-Untersuchungsanst., Hamburg). *Dtsch. LebensmittRdsch.*, 1960, **56** (5), 139–140.—About 4 medium-size oranges (or an equiv. amount of other citrus fruit) are disintegrated and, after addition of 500 ml of H_2O, 10 ml of H_3PO_4 and a little silicone anti-foam, the mixture is distilled from a 2-litre flask in a special return-flow apparatus for 2 hr. The ethereal oil, containing all the o-phenylphenol, collects in a measuring limb. A measured 1 to 2 ml of the oil is dissolved in 10 ml of light petroleum and extracted with 0·1 N NaOH (3 × 3 ml). The mixed alkaline extracts are made up to 10 ml, and aliquots (0·01 to 0·10 ml) are chromatographed (descending flow) on S. & S. 602 H/P paper, with 0·1 N NaOH as mobile phase. After 1·5 to 2 hr. the still damp paper is sprayed with a 0·5% soln. of 2,6-dibromo-p-benzoquinone-chlorimine in 96% ethanol. The blue spot given by o-phenylphenol is compared for R_F value and intensity against spots from known amounts (preferably 1 to 10 µg). Results within about 20% of theoretical values are reported.　　　　C. L. HINTON

322. Improved method for the determination of nucleic acids in yeast. Yoshiro Kuroiwa and Eiichi Kokubo. *Rep. Res. Lab. Kirin Brewery Co., Yokohama*, 1959, (2), 63–72 (in English).—The method previously described [Kuroiwa and Horie,

Ibid., 1958, (1), 33; *Bull. Agric. Chem. Soc. Japan*, 1955, **19**, 35] has been modified by introducing an extraction with water into the procedure, to ensure complete removal of interfering substances. *Procedure*—Pressed yeast (2 g, ≃ 0·5 g of dry matter) is adjusted to a moisture content of 80% in a centrifuge tube, treated with 2 g of solid CO_2 and 2 ml of ethyl ether and frozen overnight at −70°. The yeast is extracted with ice-cold ethanol (4 × 20 ml), then with ethanol - ether (3 : 1) (3 × 20 ml) at 90° for 3 min., and dried in a desiccator. The residue is then extracted with 5 ml of ice-cold water for 10 min. and centrifuged for 10 min., and this process is repeated 10 times. *Determination of RNA*—The residue is suspended in 5 ml of N $HClO_4$, stored at 0° overnight and then centrifuged. Extraction for 30 min. and centrifuging are repeated 14 times; the combined extracts are diluted to 100 ml, 1 ml is diluted to 25 ml, and the extinction is read at 260 mµ in a 1-cm cell. Then RNA phosphorus (µg per g of dry yeast) = $2500x/0·343\,w$, where x is the extinction and w is the wt. (g) of dry yeast taken. *Determination of DNA*—The residue from the RNA extraction is suspended in 5 ml of 0·5 N $HClO_4$ and extracted for 20 min. at 70°. The extraction is repeated thrice and the combined extracts are made up to 25 ml. A portion of this soln. is mixed with twice its vol. of diphenylamine reagent (Dische, *Microchem.*, 1930, **2**, 26), boiled for 5 min. and rapidly cooled, and the extinction is read after 20 min. at 600 mµ. Then DNA phosphorus (µg per g of dry yeast) = $75\,y/0·047\,w$, where y is the extinction and w is the wt. of dry yeast taken.　　　　R. E. E.

323. Rapid method for detailed analysis of the α-acid fraction of hops by gas chromatography. F. L. Rigby, E. Sihto and A. Bars (Res. Div., Canadian Breweries, Ltd., Toronto, Ont.). *J. Inst. Brewing*, 1960, **66** (3), 242–249.—The dry lead salts (250 mg) of the mixed α-acids are placed in one limb of a sealed Pyrex-glass tube bent in the form of an inverted V and pyrolysed at 400° for 10 min., the fatty acids liberated from the side-chains being condensed in the other limb. The tube is then broken at the bend, and the distilled acids are esterified by adding 0·1 ml of a soln. of 2·5 ml of conc. H_2SO_4 in 100 ml of isopropyl alcohol, sealing the tube, and heating in boiling water for 1·5 hr. The tube is then cooled and opened, the contents are shaken with 0·2 ml of H_2O and 0·1 ml of light petroleum, and an aliquot of the petroleum layer is chromatographed on a column of Celite containing 10% of Ucon 50HB2000 at 50° with a helium flow-rate of 40 ml per min. Results for 13 hop samples are compared with those obtained by counter-current distribution. Some alternative procedures are discussed.　　　　R. E. E.

324. Spectrophotometric determination of I.T.T. value of beer. Yoshiro Kuroiwa and Jun-Ichiro Kataoka. *Rep. Res. Lab. Kirin Brewery Co., Yokohama*, 1959, (2), 43–48; *Bull. Agric. Chem. Soc. Japan*, 1960, **24** (5), 511–516 (in English).—The method described avoids the uncertainty in the visual estimation of the "80% decolorisation time" of 2,6-dichlorophenolindophenol in the usual form of the Indicator Time Test (Gray and Stone, *J. Inst. Brewing*, 1939, **45**, 253). The reagent is a 0·005 M soln. prepared from very pure 2,6-dichlorophenolindophenol (cf. Heyer and Paukner, *Brauwissenschaft.*, 1952, **12**, 208). The extinction of this soln. decreases slowly with time, and it should be discarded when it cannot be completely decolorised

with ascorbic acid. *Procedure*—A "50% decolorisation standard" is prepared by mixing 0·125 ml of reagent with 10 ml of acetate buffer soln. (pH 4·4), measuring the extinction in a 1-cm cell at 520 mμ and 25°, and calculating the extinction for 80% decolorisation therefrom. The decolorisation curve of beer is obtained by adding 0·25 ml of reagent to 10 ml of beer, measuring the extinctions at given intervals of time, and plotting the results on a graph of extinction *vs.* time (in sec.). A blank curve is then obtained by taking a number of 10-ml aliquots of the beer, adding to each 0·25 ml of reagent and maintaining them at 25°, then, at given intervals, adding to the tubes, in succession, 0·05 ml of a 2% soln. of ascorbic acid in the buffer soln. and measuring the extinctions. The results are corrected by adding to each the extinction corresponding to 80% decolorisation, and the corrected results are plotted on the same graph. The time corresponding to the point of intersection of the two curves gives the I.T.T. value. The dye obeys Beer's law in beer and in water, and the ethanol in the beer has little influence on the results, which agree well with those obtained by the visual procedure.   R. E. E.

325. Colorimetric determination of iron in wine with sulphosalicylic acid. H. Konrad (Inst. f. Lebensmittelchemie beim Magistrat von Gross-Berlin). *Nahrung*, 1960, **4** (4), 365–372.—*Procedure*—The sample (10 to 25 ml) is evaporated in a platinum dish on a water bath and then ashed at 600°. The ash is dissolved in 3 ml of conc. HCl, and the soln. is diluted with H_2O and transferred to a 50-ml flask. Sulphosalicylic acid soln. (20%) (5 ml) and 0·1 g of hydroxyammonium chloride are added. A greenish-blue coloration after the addition of excess of aq. NH_3 indicates the presence of Cu, in which case a few drops of 10% KCN soln. must be added. The mixture is made up to volume, filtered if necessary, and the extinction is measured at 424 mμ; K^+, Na^+, Ca^{2+}, Cl^-, SO_3^{2-}, SO_4^{2-} and PO_4^{3-} (4 g per litre), Mg^{2+} ·(3 g per litre) and Al^{3+} (1·2 g per litre) do not interfere. Full recovery of total Fe is obtained.   I. DICKINSON

326. Chromatographic analysis of glycerides. I. Paper chromatography of synthetic and natural glycerides as their mercury addition compounds. Yoshiyuki Inoue and Manjiro Noda (Coll. of Agric., Univ., Kyoto). *J. Agric. Chem. Soc. Japan*, 1959, **33** (6), 452–456.—A new method for separating glycerides by reversed-phase paper chromatography is described. Twelve synthetic unsaturated mono-, di- and tri-glycerides containing oleic or linoleic acid are well separated as their mercuric acetate adducts on paper impregnated with petroleum hydrocarbon or tetralin. The developing solvent is methanol - acetic acid - tetralin or petroleum hydrocarbon, and the spots of the adducts are detected by spraying with a 0·2% ethanolic soln. of diphenylcarbazone. The method was applied to the separation of the triglycerides in natural fats, which give characteristic chromatograms depending upon their glyceride structure. The R_F value generally increases with increase in unsaturation of the fat.
                                           S. NATORI

327. Method for the quantitative separation of fatty acids from unsaponifiable matter. H. G. Sammons and S. M. Wiggs (Metabolic Res. Unit, Little Bromwich General Hospital, Birmingham, England). *Analyst*, 1960, **85**, 417–418.—An aliquot

of the sample (*e.g.*, food, faeces or serum) is saponified with ethanolic KOH soln. and the fatty acids and unsaponifiable matter are extracted with light petroleum. The residue from evaporation of the extract under reduced pressure at <50° is re-extracted with light petroleum and the filtered extract is reduced to a small vol. under reduced pressure. Alumina (1 g per 25 mg of fatty acid) is added, the mixture is shaken, the supernatant liquid is filtered and the residual alumina is extracted with ethyl ether. The combined extracts are evaporated and the unsaponifiable matter is weighed. The alumina is freed from solvent and is covered with methanolic HCl and set aside overnight in a stoppered flask at 37°. The methanolic liquid is filtered, the alumina is washed with ethyl ether and the solvent and HCl are removed by evaporation under reduced pressure. The residue is extracted with light petroleum, the filtered extract is dried with Na_2SO_4, then evaporated, and the methyl esters are weighed. Recovery is 90 to 95%. The method has been applied to the preparation of the methyl esters of the fatty acids in a form suitable for analysis by vapour-phase chromatography.   A. O. JONES

328. Modified method for the paper chromatography of long-chain fatty acids. R. Chayen and E. M. Linday (Dept. of Chem. Engng, University College, London). *J. Chromatography*, 1960, **3** (5), 503–504 (in English).—Long-chain fatty acid mixtures can be chromatographed directly by the reversed-phase ascending technique on Whatman No. 3 filter-paper impregnated with a 10% soln. of liquid paraffin in ethyl ether, with acetone - water (4:1) as solvent. The purity of the acetone is important. The time required for separation is about 3 hr. The spray reagent used is a mixture of 0·2% Nile blue (C.I. Basic Blue 12) sulphate soln. in ethanol (50 ml), triethanolamine (15 ml) and ethanol (50 ml); this gives a background colour of red with blue spots indicating the positions of the acids. A control sample must be run to compensate for the variations of R_F with temperature. S. M. MARSH

329. Determination of chemical antioxidants in fats after separation by partition chromatography. K. G. Berger, N. D. Sylvester and D. M. Haines (The Laboratories, J. Lyons & Co. Ltd., Kensington, London). *Analyst*, 1960, **85**, 341–345.—The chromatographic material is Silastic 181 (a synthetic rubber), with light petroleum as the stationary and aq. acetone as the mobile phase. The fat (3 to 4 g) in light petroleum soln. is applied to the column maintained at 21° by means of a water jacket. The column is washed with aq. acetone, 200 ml and then 100 ml of eluate being collected separately. In aliquots of these, butylated hydroxyanisole is determined by means of the colour produced with 2,6-dichloro-*p*-benzoquinone-4-chlorimine, the extinction being measured at 610 mμ. For determination of gallates, the eluate is treated with ferrous tartrate and sodium acetate soln. and extracted with light petroleum - isoamyl alcohol. The extinction of the extract is measured at 532 mμ for determination of the higher gallates and that of the aq. layer (diluted with acetone) at 532 mμ for propyl gallate. A test for differentiating octyl and lauryl gallates is described. To determine butylated hydroxytoluene (BHT), the soln. in 80% ethanol is treated with KOH soln., and the difference in the extinctions before and after holding the soln. at 31° for 2 hr. is a measure of the concn. of BHT.
                                           A. O. JONES

330. Antioxidants. The micro-determination of quinol. M. Z. Barakat, S. K. Shehab and A. M. Abdalla (Fac. of Vet. Med., Cairo Univ., Giza, Egypt). *J. Amer. Pharm. Ass., Sci. Ed.*, 1960, **49** (6), 360–362.—To determine quinol (I) in oils and fats, dissolve the sample (10 g or 10 ml, containing 0·5 to 2 mg of I) in ethyl ether (150 ml), add a satd. aq. soln. of NaHCO₃ (2 ml), 4% KI soln. (5 ml) and 1% starch indicator soln. (10 drops), and titrate drop by drop with 0·1% N-bromo-succinimide, with constant shaking. Use the same procedure for the determination of I in ether anaesthetics. To determine I in a photographic developer which also contains interfering sulphite, add excess of BaCl₂ soln., shake, filter and apply the procedure to the filtrate. The experimental error is $< \pm 2\%$. The method has been successfully applied to butter, lard and cod-liver oil, for amounts of I as small as 0·55 mg.   A. R. Rogers

331. Colorimetric determination of thiamine by reaction with ninhydrin. Yu. M. Ostrovskii and M. A. Gvozdeva. *Byul. Eksperiment. Biol. i Med.*, 1959, **48** (11), 120–121; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (9), Abstr. No. 11,776.—To 3 ml of phosphate buffer soln. add the sample soln. (first neutralise if necessary) containing 10 to 200 μg of thiamine and 0·5 ml of 0·1% ninhydrin soln. Place the mixture in a test-tube in a boiling-water bath for 20 min., cool, and dilute to 5 ml with water. The resulting yellow soln. has an absorption maximum in the short-wave region of the visible spectrum. Greatest sensitivity is achieved with a violet filter. It is shown that the formation of the coloured compound is due to the thiazole portion of the thiamine molecule. The free amino-group present in the thiamine pyrimidine ring probably does not take part in the reaction, since formaldehyde, which blocks the reaction of amino-groups with ninhydrin, does not affect the reaction with thiamine.   C. D. Kopkin

332. Separation of some vitamins of the B complex and ascorbic acid by thin-layer chromatography. H. Gänshirt and A. Malzacher (Wissenschaftl. Abt., Fa. Dr. Karl Thomae G.m.b.H., Biberach an der Riss, Germany). *Naturwissenschaften*, 1960, **47** (12), 279–280 (in German).—The method is applicable to about 50 to 100 μg of substance containing 1 to 10 μg of the vitamin-B group and 5 to 30 μg of ascorbic acid. Plates (20 cm × 20 cm) are coated with silica gel G (Merck) to which has been added 2% of Leuchtstoff ZS-Super (Riedel de Haen) which fluoresces at 254 mμ in u.v. light. The chromatogram is developed in the dark with acetic acid - acetone - methanol - benzene (1:1:4:14). Thiamine is strongly adsorbed by the plate, the other vitamins have the following R_F values—pyridoxine 0·15, ascorbic acid 0·30, riboflavine 0·35, Ca pantothenate 0·57, nicotinamide 0·65, and biotin 0·80. Methods for the identification of the individual vitamins are given.   B. Heining

333. Determination of vitamin B₁₂. A critical review. W. H. C. Shaw and C. J. Bessell (Glaxo Lab. Ltd., Greenford, Middx., England). *Analyst*, 1960, **85**, 389–409.—The physical and chemical methods reviewed include colorimetric methods, direct and indirect spectroscopic methods and applications of polarography and isotope dilution. It is suggested that spectrofluorimetric methods would repay further study. Methods for separation from crude materials and from analogues include adsorption chromatography, column and paper partition chromatography ion-exchange methods and paper electrophoresis. Methods for pre-treatment of natural materials (*e.g.*, liver, blood and urine) in which the B₁₂ may be bound are described. Microbiological assays, assays with higher animals and clinical assays are reviewed. (174 references.)   A. O. Jones

See also Abstracts—**157**, Ethanol in aq. media. **306**, Potency of isomerised vitamin-A palmitate.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

334. Detection and estimation of low concentrations of aldehyde in air. E. E. Hughes and S. G. Lias (Nat. Bureau Standards, Washington, D.C., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 707–708.—The aldehyde is adsorbed from the air stream on a column of silica gel, which is then treated with a mixture of p-phenylenediamine (I) and H₂O₂. The oxidation of I is catalysed by the aldehyde and the depth of the colour formed is a measure of the aldehyde adsorbed. Concn. as low as 10^{-8} mole % can easily be detected. Nitriles, aldehyde ammonia, oximes and aldehyde bisulphite compounds interfere, but these are not usually significant air pollutants.   G. P. Cook

335. Rapid determination of toluene and styrene vapours in the atmosphere. A. J. Blake and B. A. Rose (D.S.I.R., Lab. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1960, **85**, 442–445.—The apparatus is that previously described for the determination of benzene vapour ("Methods for the Determination of Toxic Substances in Air, No. 4: Benzene Vapour," H.M. Stationery Office, London, 1958). For determination of toluene vapour the bubbler contains 0·5 ml of formaldehyde soln. (40% w/v) diluted to 10 ml with H₂SO₄ - H₃PO₄. The number of inflations of the bulb required to produce a colour in the reagent equal to that in the standard soln. is noted and the toluene concn. (p.p.m.) is ascertained from a table. For the determination of styrene by a similar procedure, the bubbler contains 10 ml of 89·5% H₂SO₄. The standard colour soln. for toluene is a mixture of 64·3 ml of 0·1 M CoSO₄, 1·7 ml of M/60 K₂Cr₂O₇ and 34·0 ml of 0·125 M (NH₄)₂SO₄.NiSO₄, and for styrene 6·9 ml of M/60 K₂Cr₂O₇ and 4·3 ml of 0·1 M CoSO₄ mixed and diluted to 250 ml. The reagent for determining toluene is comparatively insensitive to benzene and 100 p.p.m. of styrene produces no colour after six inflations of the bulb. Benzene, toluene and dimethylstyrene (100 p.p.m. of each) produce no colour in the test for styrene, but vinyltoluene interferes.   A. O. Jones

336. A review of the literature of 1959 on waste water and water-pollution control. Analytical methods. Water Pollution Control Fed. *J. Wat. Pollut. Control Fed.*, 1960, **32** (5), 443–452.—A review is presented of methods for determining B.O.D., C.O.D., dissolved oxygen, nitrogen compounds, sulphur compounds, halogen compounds, organic compounds and metal ions in water. (93 references.)   C. A. Slater

337. Application of cellulose phosphate to the testing of high-purity water and the development of a spot test of high sensitivity [for copper and iron]. N. F. Kember (National Chem. Lab., Teddington, Middx., England). *Analyst*, 1960, **85**, 449–451.— In the apparatus described, a disc of cellulose phosphate is held in a polyethylene block surmounted by a glass funnel. For the determination of Cu, the pH of the sample is adjusted to >4 with dil. aq. NH_3 and the liquid is passed through the disc. The disc is removed, placed on filter-paper, exposed to NH_3 vapour and sprayed with a 0·5% soln. of dithio-oxamide in 80% (v/v) aq. ethanol. The colour produced is compared with that of previously prepared standards. For the determination of Fe, the sample is acidified with 1% (v/v) HCl and poured through the disc, which is then dipped into $K_4Fe(CN)_6$ soln., set aside for 2 min. on filter-paper, washed with water and compared with previously prepared standards. The lower limits of detection are $0·4 \times 10^{-9}$ part of Cu and 2×10^{-9} part of Fe in a 50-ml sample.

A. O. JONES

338. Chromatographic separation and identification of organic acids. Application to detection of organic acids in river waters. H. F. Mueller, T. E. Larson and M. Ferretti (Illinois State Water Survey, Urbana, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 687–690.—The sample is evaporated to low bulk in the presence of NaOH and after acidification the organic acids are extracted into ether. The acids are titrated with NaOH soln., the ether is removed by evaporation and the remaining aq. phase is evaporated to 1 ml, acidified, and chromatographed on a silicic acid column; the mobile phase consists of various concn. of n-butanol in $CHCl_3$, and dil. H_2SO_4 is the stationary phase. The 5-ml fractions are analysed by titration with NaOH soln., with bromothymol blue as indicator. The recoveries are >90% for all acids. The identity of some acids is confirmed by comparison of the R_F values obtained for pure acids by paper chromatography of the sodium salts, free acids or hydroxamates. Results for 25 samples taken from the Mississippi and Ohio rivers are discussed.   G. P. COOK

339. Determination of free and total amino-acids and amines in waste waters [from sugar refineries]. S. Jankovic (CEBEDEAU, Liège, Belgium). *Bull. Cent. Belge Étud. Docum. Eaux*, 1960, (113), 165–169.—Two colorimetric methods are described. *Procedure*—Evaporate 100 ml of sample, under reduced pressure and below 40°, to a vol. of 10 ml. Then (*i*), hydrolyse 5 ml of the concentrate with 5 ml of conc. HCl in a sealed tube at 105° for 24 to 48 hr. Dry over P_2O_5 and NaOH *in vacuo*. Dissolve the residue in H_2O, centrifuge at 1200 r.p.m., and make the supernatant liquid up to a vol. such that 1 ml contains 10 μg of N. Determine the total amine N at 485 mμ with Na 1,2-naphthaquinone-4-sulphonate (**I**) (Frame *et al.*, *Actualités Biochim.*, 1946, **4**, 38). (*ii*) Transfer 1 ml of concentrate to a centrifuge tube, add 10 ml of 95% ethanol to precipitate proteins and centrifuge at 1200 r.p.m. Take an aliquot containing 2·5 to 10 μg of N and determine the free α-amino-acids at 570 mμ with ninhydrin (Yamagishi and Yoshida, *J. Pharm. Soc. Japan*, 1953, **73**, 675). Free amines and amines from decarboxylated amino-acids are extracted by continuous liquid - liquid extraction for 24 to 48 hr. with ethyl ether and determined with **I**. Beer's law is obeyed over the range 10 to 30 μg of N with method (*i*) and 2·5 to 10 μg with method (*ii*).

R. A. HENDEY

340. Determination of cobalt in water. I. A. Mikhalyuk (Kiev Med. Inst.). *Lab. Delo*, 1960, **6** (3), 26–27.—For the calibration graph prepare 10-ml aliquots containing Co (as chloride or nitrate) at intervals of 0·2 μg in the region of the expected value. To each of these aliquots add H_2O (10 ml), HNO_3 (1 : 1) (0·5 ml) and HCl (1 : 1) (0·5 ml). Boil for 90 sec., add 1 ml (≡ 1 mg) of nitroso-R-salt soln. and 2 ml of fresh Na acetate soln. Boil for 45 sec., add HNO_3 (1 : 1) (2 ml) and boil for a further 45 sec. Cool and adjust the vol. to 20 ml with H_2O. Measure the extinction with a blue filter. Evaporate 1 litre of sample to dryness in a porcelain dish. Moisten the residue with H_2O, dissolve it in conc. HNO_3 (2 ml), evaporate to dryness and proceed as described above. The accuracy is within 7%.

A. BURWOOD-SMITH

341. Possible use of ammonium 12-molybdo-phosphate for assaying certain radioactive fission products in water. R. W. C. Broadbank, S. Dhaba-nandana and R. D. Harding (School of Chemistry, Coll. of Technol. and Commerce, Leicester, England). *Analyst*, 1960, **85**, 365–370.—Microgram amounts of many cations, including those of the fission products ^{137}Cs - ^{137m}Ba, ^{90}Sr - ^{90}Y and ^{144}Ce - ^{144}Pr, are removed from neutral soln. by passage through a pre-formed layer of ammonium 12-molybdophosphate. Caesium is selectively retained by this compound from soln. in dil. HNO_3. To determine ^{137}Cs in rain water, the sample, acidified with HNO_3, is poured through an even layer of $(NH_4)_3PO_4.12MoO_3$ on Whatman No. 1 filter-paper in a de-mountable Buchner funnel of diameter 1 in. The filter, dried by suction, is mounted with the ppt. on an aluminium tray and counted with an end-window Geiger counter. For μg amounts of Cs no correction for self-absorption appears to be necessary. In a quoted application of the method, 1100 ml of rain water, collected in 1958, showed an activity of 12 ± 2 μμC per litre. Only relatively large amounts of Tl+ appear to affect the removal of Cs from dil. HNO_3 soln. If present in sufficient amount Ca and Mg interfere with the assay of ^{90}Sr - ^{90}Y by this procedure, even in the presence of EDTA. Directions for the preparation of the layer of $(NH_4)_3PO_4.12MoO_3$ are given.

A. O. JONES

342. Studies on fluorescence of coumarin derivatives. I. Fluorimetric analysis of warfarin. Yoji Ichimura (Nat. Hygiene Lab., Tamagawayoga-machi, Setagayaku, Tokyo). *J. Pharm. Soc. Japan*, 1959, **79** (8), 1079–1082.—Warfarin was determined by means of its fluorescence in the u.v. region. The fluorescence was strongest, and fairly stable, in alkaline ethanolic soln. (pH 10) and the response was linear over the range 0·5 to 8 μg per ml.

S. NATORI

See also *Abstracts*—**37**, **38**, Be in air. **120**, F⁻ in water. **382**, Detection of contaminants in air by gas chromatography.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

343. Leaf analysis as a guide to the nutrition of fruit crops. I. General principles, sampling techniques and analytical methods. C. Bould, E. G. Bradfield and G. M. Clarke (Dept. of Agric. and

Hortic., Long Ashton Res. Sta., Bristol, England). *J. Sci. Food Agric.*, 1960, **11** (5), 229–242.—The concept of critical nutrient levels is discussed and variations in the chemical composition of leaves due to other factors such as age and season are illustrated. In a statistical study of a particular sampling procedure, the total coeff. of variation was about 6%, of which 1·2% was due to the analytical stage. After digestion of a dried sample with H_2SO_4, Na_2SO_4 and Se, N was determined as NH_4^+, P as molybdovanadophosphate and K by flame photometry. On a further sample, digested with HNO_3 - $HClO_4$, Ca (and K) were determined by flame photometry, and Mg with Titan yellow after removal of interfering ions by ion exchange.

P. D. PARR-RICHARD

344. Wet-combustion apparatus and procedure for organic and inorganic carbon in soil. L. E. Allison (U.S. Salinity Lab., Riverside, Calif.). *Proc. Soil Sci. Soc. Amer.*, 1960, **24**, 36–40.—An apparatus is described in which the CO_2 evolved from the sample on acidification, oxidation and boiling under reflux is carried in a stream of air through KI soln., Ag_2SO_4 soln., conc. H_2SO_4, granulated zinc and anhydrone, and absorbed in a self-indicating absorbent and weighed. The procedure can be applied to saline soils high in Cl^- and to extracts that can be evaporated to dryness. If a blank determination yields >1 mg of CO_2 the sample should be pre-aerated. *Procedures*: (*i*) *Organic C in non-carbonate soils*—The sample is treated with conc. H_2SO_4 - 85% H_3PO_4 (3:2) and solid $K_2Cr_2O_7$, heated for 10 min. with aeration, then aerated for a further 10 min. (*ii*) *Organic C in carbonate soils*—The sample is boiled with acid $FeSO_4$ soln. (57 ml of conc. H_2SO_4 and 92 g of $FeSO_4.7H_2O$ made up to 1 litre with water), then cooled, and treated as in (*i*). (*iii*) *Organic C in soil extracts*—The extract is treated with acid $FeSO_4$ soln. and evaporated to dryness in the digestion flask, then treated as in (*i*). (*iv*) *Inorganic C in calcareous soils*—The dry sample is treated with acid $FeSO_4$ soln., boiled for 3 min. with aeration and then aerated for a further 10 min. The methods are rapid and accurate.

R. E. E.

345. Determining strontium-90 in soil by electro-dialysis and ion-exchange method. E. R. Graham (Sci., Lab., Los Alamos, N. Mex., U.S.A.). *Soil Sci.*, 1959, **88**, 11–15.—The sample (5 g or more) is placed in a membrane sac (usually filter-paper), covered with water and electrodialysed at 100 V for 12 hr. or more with a platinum-foil anode and stainless-steel cathode. The liquid, together with washings with M HCl, is treated with sufficient EDTA to make the soln. 1%, stirred and adjusted to pH 10 with NaOH. Complete chelation of Ca and Mg is checked with Eriochrome black T. The soln. is then adjusted to pH 5·5 to liberate ^{90}Sr and passed through a column of Dowex 50-X12 resin (activated by alternate washings with 5% HCl and 5% NaCl soln. followed by washing with water till free from excess of NaCl) at a rate of 8 ml per sq. cm per min. The existing ^{90}Y is then eluted with 500 ml of a soln. containing 1% of citric acid and 0·75% of EDTA, adjusted to pH 5; the completion of this elution is taken as zero time for the development of further ^{90}Y activity on the column. After a given time-interval (48 hr. or more) the ^{90}Y formed is eluted at the above rate, first with 50 ml of 5% citric acid soln. at pH 1·9 to 2·1, and then with 350 ml of 5% citric acid soln. adjusted to pH 3·8. The combined eluates are

evaporated to 200 ml, treated with 1 ml of carrier soln. (30 mg of Y per ml) and sufficient solid KF to make the soln. 3 M, and the ppt. is collected on a tared filter, dried with ethanol and ethyl ether, weighed and counted. The result is referred to a graph of time *vs.* ^{90}Y activity as a percentage of the total ^{90}Sr activity.

R. E. E.

346. Radiometric determination of potassium in NPK fertilisers. K. Martínek and J. Gottfried (Res. Inst. Inorg. Chem., Ústí Nad Labem, Czechoslovakia). *Chem. Průmysl*, 1960, **10** (6),300–302.—The determination is based on the measurement of the natural radioactivity of ^{40}K and results were in good agreement with those obtained gravimetrically with Na tetraphenylborate, or with those by the flame-photometric method.

J. ZÝKA

347. Polarographic determination of thiourea. V. Medonos and M. Vávrová (Inst. Chem. Technol., Prague). *Chem. Průmysl*, 1960, **10** (5), 234–237.—Thiourea yields a polarographic wave in N H_2SO_4. The intensity of the limiting current between the dropping-mercury electrode and the S.C.E. in the range $-0·2$ to $-0·5$ V is linearly dependent on the concn. of thiourea (2×10^{-3} N to 6×10^{-3} N). Cadmium acetate (3 g per litre) is added to the supporting electrolyte to remove sulphides. Cyanamide, dicyanamide and urea do not interfere. The measured values are referred to a calibration curve. If a 0·1 N mercurous sulphate reference electrode is used, the determination can be carried out without the use of an external voltage. The average error is ±2%. The method may be used for the determination of thiourea in calcium cyanamide.

J. ZÝKA

348. Determination of nitrofurazone in feed mixtures. A. H. J. Cross, R. A. Hendey and S. G. E. Stevens (Smith Kline and French Lab. Ltd., Welwyn Garden City, Herts., England). *Analyst*, 1960, **85**, 355–359.—The sample ($\equiv \simeq 1$ mg of nitrofurazone) is extracted with light petroleum in a Soxhlet apparatus, the extract being rejected. The dry residue is extracted with CCl_4, the extract being again rejected. The final residue is now extracted with acetone in an apparatus protected from light. Most of the acetone in the extract is removed, 0·1 N $KMnO_4$ is added until the colour persists, the remainder of the acetone is removed, the yellowish residue is dissolved in dimethylformamide and the soln. is adjusted to a known vol. Each of two equal aliquots ($\equiv 0·05$ to 0·35 mg of nitrofurazone) is treated with 5 ml of a 5% soln. of phenol in dimethylformamide, then one is treated with 2·5 ml of N NaOH and diluted to a known vol. with dimethylformamide, and the other (the blank) with a soln. of sodium dithionite in N NaOH and diluted with dimethylformamide. After centrifuging the soln., the extinction at 530 mμ of the sample soln. is measured against the blank and referred to a calibration graph. Recovery of known amounts from complex feed mixes ranged from 99 to 101·5%.

A. O. JONES

349. Determination of captan in plant extracts. E. Somers and D. V. Richmond (Res. Sta., Long Ashton, Bristol, England). *Analyst*, 1960, **85**, 440–442.—A suitable aliquot of a soln. of captan in benzene is evaporated almost to dryness on a water bath in a stream of dry air, the residue being allowed to dry at room temp. Pyridine - tetraethylammonium hydroxide soln. (prep. described) is added and

after 3 min. the extinction of the liquid is measured at 430 mμ and referred to a calibration graph. Beer's law is obeyed over the range 0 to 75 μg of captan. gamma-BHC, chlorobenzilate, chlorbenside, DDT, dieldrin and endrin do not interfere. With fruit and leaf extracts, recovery is satisfactory except in the presence of high concn. of plant wax (*e.g.*, with strawberries).   A. O. JONES

See also Abstract—**351**, Soil percolator.

5.—GENERAL TECHNIQUE AND APPARATUS

General

350. Simple and universally applicable technique for handling air- and moisture-sensitive substances. T. Niedermaier (Badische Anilin- & Soda-Fabrik A.-G., Ludwigshafen/Rhein, Germany). *Z. anal. Chem.*, 1960, **174** (6), 407–411.—The weighing bottle is kept in a stream of Ar or of specially purified N in a cylindrical vessel, and the sample is introduced by means of a tube sealed into the stopper of the cylinder. If liquid, it is forced over under pressure and, if solid, it is tipped in from a modified filter-flask. The bulk sample is maintained in the inert atmosphere. The weighing bottle is then closed by a ground-glass stopper on a long stem, and is readily lifted out of the cylinder by means of a platform on a stem passing through a sleeve in the bottom of the cylinder. The apparatus is illustrated.   J. P. STERN

351. An all-round soil percolator. K. Gundersen (Botanical Inst., Univ. Gothenburg, Sweden). *Science*, 1960, **132**, 224–225.—A description is given of a simple soil percolator for studies of the soil in a water-saturated state under aerobic conditions. The apparatus can also be used in a closed system with a gas other than air for anaerobic experiments, and the whole percolator can be autoclaved easily for experiments with sterile soil.   R. A. HENDEY

352. Fractionating column for analysis of nucleic acids. J. D. Mandell and A. D. Hershey (Carnegie Inst. of Washington, Cold Spring Harbor, N.Y.). *Anal. Biochem.*, 1960, **1** (1), 66–77.—The column is prepared with kieselguhr and serum albumin (*cf.* Lerman, *Biochim. Biophys. Acta*, 1955, **18**, 132), the albumin being made basic by methylation with methanol. Nucleic acids are adsorbed by the column from neutral soln. 0·4 M or less in NaCl, and are eluted by higher salt concn. that are characteristic for the individual nucleic acids. Applications are described.   H. F. W. KIRKPATRICK

353. Alignment block and clamp for capillary viscometers. W. M. Robinson (BP Australia, Ltd., Birkenhead, S. Australia). *Bull. A.S.T.M.*, 1960, (245), 69–70.—Constructional details are given of an arrangement which, fitted to Cannon-Fenske viscometers, reduces the max. deviation of the calibration const. (*C*) to $\pm 0.1\%$ (for opaque liquids) and $\pm 0.0008\%$ (for clear liquids). The holder can be adapted to any other type of viscometer.   W. J. BAKER

354. Pyrolysis as an analytical tool. R. N. Rogers, S. K. Yasuda and J. Zinn (Los Alamos Sci. Lab., N. Mex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 672–678.—Pyrolysis curves of both organic and inorganic compounds are approximately the first derivative of the thermogravimetric pyrolysis curves and a new, simple type of instrument, designed to replace the thermobalance, is described. The way in which pyrolysis data may be used to obtain kinetic constants is discussed and several pyrolysis curves are presented.   K. A. PROCTOR

355. Thermochemical titrations in fused salts. J. Jordan, J. Meier, E. J. Billingham, jun., and J. Pendergrast (Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 651–655.—Chloride has been determined at concn. between 8×10^{-4} and 2×10^{-2} M in a molten $LiNO_3$ - KNO_3 eutectic at 158° by a thermometric precipitation method, with a standard soln. of $AgNO_3$ in the eutectic as titrant. The titrations were performed automatically by remote control in an adiabatic cell operated at 150° to 200°, in which random temp. fluctuations were reduced to $\pm 0.0005°$. Temperature changes during the titration were monitored by means of a thermistor bridge and automatically recorded. The method appears to be generally applicable to the rapid and convenient determination of chloride in fused nitrate melts.   K. A. PROCTOR

See also Abstracts—**14**, High-temp. vacuum furnace for thermal analysis. **151**, Apparatus for H in hydrocarbons.

Chromatography, ion exchange, electrophoresis

356. An extension of the gradient-elution principle. D. G. O'Sullivan (Courtauld Inst. Biochem., Middlesex Hosp. Med. School, London). *Analyst*, 1960, **85**, 434–440.—Methods of eluting chromatographic columns with more than one solvent are discussed mathematically. In the "constant volume" method, as each drop of the first solvent falls on the column from the reservoir an equal drop of the second falls into the reservoir. For greater flexibility in this type of concn. gradient, the second solvent is added to the first at a rate different from that at which the first solvent feeds the column. This can be effected by connecting two cylindrical reservoirs of different cross-sectional area by a siphon, and arranging the flow to the column so that the levels of liquid in the two reservoirs fall at equal rates. Both the constant-volume method and the siphon method are extended to three eluting solvents and, in addition to the mathematical treatment, graphs of the concn. of the second and third solvents in the mixed eluting liquid plotted against the volume of eluting liquid leaving the mixing apparatus are shown.   A. O. JONES

357. Technique for simultaneous multiple-column gradient-elution chromatography. P. Vestergaard (Rockland State Hospital, Orangeburg, N.Y., U.S.A.). *J. Chromatography*, 1960, **3** (6), 560–569.—A variable-speed drive operates a syringe which delivers one liquid to a mixing chamber. This feeds eluent to a number of columns in parallel so that the same gradient is used for a set of standards

and unknowns. The gradient can be changed during a run by altering the speed of the syringe drive.   A. R. ROGERS

358. Optimisation of resolution - time ratio with packed chromatographic columns. R. J. Loyd, B. O. Ayers and F. W. Karasek (Res. and Devel. Dept., Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 698–701.—The effects of the quantity of stationary phase and the choice of carrier gas on the resolution - time ratio obtained with packed gas-chromatographic columns have been investigated. Considerable improvement in the time required for a separation can be achieved by using a small proportion of stationary phase and a carrier gas of low viscosity and high diffusivity.
                                K. A. PROCTOR

359. A multicollector: a fraction collector for the simultaneous collection of fractions from a number of chromatographic columns. P. Vestergaard (Rockland State Hospital, Orangeburg, N.Y., U.S.A.). *J. Chromatography*, 1960, **3** (6), 554–559.— The collector (described and illustrated) is inexpensive and easy to build.   A. R. ROGERS

360. Automatic recording elution analyser for organic acids. Chie Yamazaki, Nobuya Nagashima and Tadao Takenishi (Central Res. Lab., Ajinomoto Co., Inc., Kawasaki, Kanagawa, Japan). *Anal. Chem.*, 1960, **32** (6), 733–734.—In the method described, the effluent from an elution-chromatographic column flows continuously through a 1-mm cell in an i.r. spectrograph and the extinction at 1715 cm^{-1} is recorded. Modifications to a Perkin - Elmer spectrophotometer Model 21 to enable it to be used for this purpose are described.
                                K. A. PROCTOR

361. Apparatus for collecting and drying organic acid fractions. J. H. Schwartz (Eastern Reg. Lab., Agric. Res. Service, U.S. Dept. of Agric., Philadelphia, Pa.). *J. Chromatography*, 1960, **3** (5), 491–493 (in English).—The attachment described can be used on a Technicon fraction collector for collecting organic acid fractions that have to be subsequently evaporated and titrated. The rack holds $25 \text{ mm} \times 50 \text{ mm}$ shell vials, which are more suited to the purpose than test-tubes, and the method of construction is detailed. Also described are special racks holding 10 vials which can be fastened together to form larger units and then placed in a special drying chamber for evaporation.   S. M. MARSH

362. PTFE and glass pump: chromatographic applications. A. C. Arcus (Nutrition Res. Dept., Med. Sch., Dunedin, New Zealand). *J. Chromatography*, 1960, **3** (5), 411–414 (in English).—A detailed description is given of a pump with a glass cylinder and PTFE (polytetrafluoroethylene) piston. The piston has a sealing ring which ensures that no solvent leaks back. Chromatographic applications include the supplying of solvent under pressure or at constant rate, the de-gassing of solvents and gradient elution.   S. M. MARSH

363. A new apparatus for paper-chromatographic analyses in an inert atmosphere. P. Szarvas, T. Balogh and L. Máczay. *Magyar Kém. Foly.*, 1960, **66** (4), 137–139.—The separation of Ti^{III} from Ti^{IV} and of V^{II} from V^{III}, V^{IV} and V^{V} has been studied. As Ti^{III} and V^{II} are readily oxidised in air, the procedure is carried out in an apparatus (described

and illustrated) in which the preparation of the lower-valency ion, its application to the paper, and the drying and development of the chromatogram are carried out in an inert atmosphere. The reduction is performed by means of nascent hydrogen in a Jones-type reductor that forms part of the dropping device for loading the chromatogram.
                                R. E. E.

364. Radial-development chromatography. II. L. Peyron (Lab. de Recherches, Éts. A. Chiris, Grasse, France). *Bull. Soc. Chim. France*, 1960, (6), 1243–1247.—This bibliography of publications, mainly from 1957 onwards, is subdivided according to (*i*) elution procedures, and (*ii*) separation of organic and inorganic compounds. A few of the references are annotated briefly. The usefulness of radial chromatography in the analysis of essential oils and their constituents is stressed. (*Cf. Anal. Abstr.*, 1959, **6**, 1578.)   (222 references.)
                                W. J. BAKER

365. Improved Zaffaroni-type solvent system for paper chromatography. J. C. Touchstone and M. Kasparow (Dept. of Obstetrics and Gynecology, Sch. Med., Univ. Pennsylvania, Philadelphia). *Anal. Biochem.*, 1960, **1** (1), 91–93.—A solvent system consisting of methylcyclohexane and dimethylformamide, suitable for the separation of steroids not resolved by other systems, is described. Details are given of the separation of 17-hydroxydeoxycorticosterone from corticosterone and from 11-deoxycorticosterone.   R. A. BRENAN

366. An apparatus for elution from paper chromatograms. M. J. Canny (Botany School, Cambridge, England). *J. Chromatography*, 1960, **3** (5), 496–497 (in English).—The apparatus can be used for eluting large pieces of paper with small volumes of volatile solvents, and consists essentially of a reservoir with a paper wick, inside a glass tube which holds the paper to be eluted. The assembly is cheap and quickly constructed, only the minimum of supervision is necessary and large numbers of papers can be eluted simultaneously.
                                S. M. MARSH

367. Automatic solvent-addition funnel for paper chromatography. B. L. Van Duuren (Dept. of Ind. Med., New York Univ., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 732–733.—The funnel described makes it possible to start the running of a chromatogram automatically after a pre-determined equilibration period, and can be used for both ascending and descending chromatography.   K. A. PROCTOR

368. Paper chromatography: multiple-spot formation by pure substances. A. H. Beckett, M. A. Beaven and A. E. Robinson (Chelsea Coll. Sci. and Technol., London). *Nature*, 1960, **186**, 775–776.— A study of amines and amine salts, with acid and neutral solvent systems, has shown that double-spot formation is dependent on the relative R_F values of individual acids and bases of the salt, the relative concn. of the acid and base, and the relative dissociation constants of the acids present in the salts and in the solvent system. The conditions for multiple-spot formation are likely to occur with amines in biological extracts that contain organic acids, or to which trichloroacetic acid has been added, and in the investigation of the metabolism of basic drugs if strongly acid compounds are also products of metabolism.   A. C. R. HARTLEY

369. **Rapid estimations of zone content in paper chromatographs. VI. R.LF. method.** G. G. Blake (Dept. of Chem., Sydney Univ., Australia). *Anal. Chim. Acta*, 1960, **22** (6), 546–548 (in English).— The zone is cut out and extracted with a suitable solvent (*e.g.*, water) and the conductance is measured by a rectified low-frequency (R.LF.) method (*Ibid.*, 1955, **13**, 394). The amount of solute present is read from a calibration curve prepared from standard soln. The suitable range of solute concn. is 0·002 N to 0·00016 N, but for solute concn. <1 part in 10⁴ the electrostatic discharge method (*Ibid.*, 1959, **21**, 545) is preferred. R. M. S. HALL

370. **Certain aspects of bromophenol blue staining deduced from spot tests on filter-paper.** G. G. Selman (Inst. of Animal Genetics, Edinburgh, Scotland). *J. Chromatography*, 1960, **3** (6), 531–535.— Different ways of using bromophenol blue for staining proteins on filter-paper have been compared. A positive reaction may be given by thiols, thiamine and some imidazole derivatives. A. R. ROGERS

371. **Spotting apparatus for the application of large quantities of fluids on paper chromatograms.** J. Logothetis (Univ. of Minnesota Med. Sch., Minneapolis, U.S.A.). *J. Chromatography*, 1960, **3** (5), 488–491 (in English).—The apparatus transfers fluid specimens from eight syringes by continuous slow ejection on to chromatographic paper while the air pressure is reduced under the spotting area by an adjustable vacuum-pump device; this ensures rapid evaporation of the drops to give deposits of dry material covering a small uniform area. Full constructional details are given, with diagrams. S. M. MARSH

372. **Quantitative radioassay of paper chromatograms by liquid scintillation counting. Application to carbon-14-labelled salicylic acid.** W. F. Bousquet and J. E. Christian (Bionucleonics Dept., Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 722–723.—The procedure described is valid for the quant. assay of ¹⁴C-labelled salicylic acid on paper chromatograms. An acid scintillator system is used and eliminates the spurious count rates observed in non-acid scintillation media. K. A. PROCTOR

373. **Isolation and determination of volatile compounds by adsorption on charcoal.** J. H. Dhont and C. Weurman (Central Inst. for Nutrition and Food Res. T.N.O., Utrecht, The Netherlands). *Analyst*, 1960, **85**, 419–422.—The apparatus used is described. The vapour of the sample is conveyed by a current of air into a weighed amount of charcoal, which is then re-weighed and placed on the top of a charcoal column. The steam-jacketed column is heated to 100° and dried by passage of a stream of N. The stream of N is then saturated with dioxan vapour and directed through the column into the katharometer. Desorption is followed by the galvanometer, the first deflection indicating the entrance of the displaced compound and the second the break-through of the displacing dioxan. The sample vapour is condensed in a U-tube cooled with dry ice and acetone, its vol. is measured at room temp., and an aliquot is analysed by vapour-phase chromatography, a 3-metre column of Carbowax 1540 at 70° being used and the results plotted by an automatic recorder. The ratio of the peak height for dioxan to the peak height of the desorbed compound is calculated and compared with those on chromatograms of mixtures of known amounts of dioxan and the compound being investigated. A. O. JONES

374. **Portable carbon dioxide-conversion apparatus for gas - liquid chromatography.** M. C. Simmons, L. M. Taylor and M. Nager (Houston Res. Lab., Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 731–732.—A compact, portable CO_2-conversion unit for attachment to gas chromatographs is described. It allows the separated components to be detected as hydrocarbon bands which can be trapped for positive identification or more detailed analysis by other methods. Alternatively, hydrocarbons emerging from the chromatograph may be directed into the conversion unit, converted into CO_2, and detected with a separate thermal-conductivity detector. K. A. PROCTOR

375. **Coiled columns and resolution in gas chromatography.** J. C. Giddings (Dept. of Chem., Univ. of Utah, Salt Lake City, U.S.A.). *J. Chromatography*, 1960, **3** (6), 520–523.—With certain assumptions it is shown that the increase in plate height due to bending is equal to $7vr^4/48R^2D$, where v is the linear gas velocity, r is the tube radius, R is the radius of curvature, and D is the coeff. of lateral diffusion. The effect is marked with preparative-scale columns. A. R. ROGERS

376. **Simple accessory to a commercial gas chromatograph for micro-catalytic studies.** L. S. Ettre and N. Brenner (The Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *J. Chromatography*, 1960, **3** (6), 524–530.—The gas-sampling valve of a commercial gas chromatograph is replaced by a combined sampler and micro-reactor. This makes possible the analysis of the products of either periodic or flow-type reactions. A. R. ROGERS

377. **Relative detector response in gas chromatography. [I.]** G. R. Jamieson (Chem. Dept., Paisley Tech. Coll., Scotland). *J. Chromatography*, 1960, **3** (5), 464–470 (in English).—The relative response of thermal-conductivity detectors to a wide range of organic compounds has been investigated, with N as the carrier gas. Three comparison compounds were chosen, *viz*, benzene up to 95°, *p*-cymene at 160° and methyl palmitate at 210°. Tabulated results show that there is an increase in relative response with increasing molecular weight through a homologous series, and that for isomeric compounds there is a decrease in relative response with an increase in chain-branching. Relative response does not vary if the flow rate of N is varied within narrow limits, but is temperature-dependent. As more data become available, relative detector response might be used in conjunction with retention-volume data for identification purposes. S. M. MARSH

378. **Effect of an argon - nitrogen carrier-gas mixture on the sensitivity of a gas-chromatographic ionisation detector.** D. Welti and T. Wilkins (Unilever Ltd., Food Res. Dept., Colworth House, Sharnbrook, Bedford, England). *J. Chromatography*, 1960, **3** (6), 589–591.—The sensitivity of the "argon detector" can be varied continuously over a wide range by use of a mixture of Ar and N as carrier gas, and a conventional detector can be used with preparative-scale columns. The greatest decrease in sensitivity occurs as the proportion of N is increased from 0 to 10%. Flow-rate settings are critical for good reproducibility. A. R. ROGERS

379. **Thermal conductivity cell response and its relationship to quantitative gas chromatography.** R. L. Grob, D. Mercer, T. Gribben and J. Wells (Chem. Dept., Wheeling Coll., W. Va., U.S.A.).

J. Chromatography, 1960, **3** (6), 545–553.—Analysis of blends of alcohols has shown that experimentally measured response values should be used to relate peak areas with wt. of components; the errors are <1% but, if the relative areas are used as a measure of wt. (%) on the assumption of direct proportionality, the errors are as high as 10%.

A. R. ROGERS

380. Compensated Wheatstone's bridge circuit for gas-chromatographic katharometry. A. B. Littlewood (Dept. Chem., Kings Coll., Univ. of Durham, Newcastle upon Tyne, England). *J. Sci. Instrum.*, 1960, **37** (6), 185–188.—Suitable shunt and series resistances included in the katharometer arms of the Wheatstone bridge circuit considerably reduce base-line drift caused by imperfect temp. or voltage control. Equations are given from which the values of these resistances can be calculated from the observed drift in their absence.

G. SKIRROW

381. Response of the β-ray ionisation detector to unesterified lower fatty acids in gas - liquid chromatography. C. J. F. Böttcher, G. F. G. Clemens and C. M. van Gent (Dept. of Phys. Chem., Univ., Leiden, Netherlands). *J. Chromatography*, 1960, **3** (6), 582–584.—The relative peak areas per unit mass of the straight-chain fatty acids (from C_4 to C_{10}) increase with molecular weight up to about 150. The relative peak area per mole is a linear function of the log of the number of carbon atoms.

A. R. ROGERS

382. A Tesla discharge detector for gas chromatography. J. C. Sternberg and R. E. Poulson (Kedzie Chem. Lab., Michigan State Univ., East Lansing, U.S.A.). *J. Chromatography*, 1960, **3** (5), 406–410 (in English).—A laboratory Tesla coil leak tester has been used in the construction of two simple cheap high-sensitivity detectors for gas chromatography. Variations in gas composition may be detected by measuring either the emitted light intensity, or a direct-current signal produced by inserting a pair of probe-electrodes asymmetrically into the discharge. Both means of detection can be incorporated into a single detector. Air may be used as the carrier gas, and the detector could be employed for the measurement of humidity and vapour pressures above solvents or solutions, mol. wt. determinations, and the detection of atmospheric contaminants.

S. M. MARSH

383. An apparatus for paper ionophoresis. S. Lovett (Microbiol. Res. Estab., Porton, Wilts., England). *Chem. & Ind.*, 1960, (25), 709–710.—The apparatus described has been designed to overcome the staining obtained when copper-wire electrodes are used in electrophoresis. Provision has also been made for the simultaneous development of several electropherograms.

K. A. PROCTOR

384. Simple apparatus for the application of specimens in continuous paper electrophoresis. W. Matthias (Inst. f. Pflanzenzüchtung, Dtsch. Akad. der Landwirtschaftswissenschaften, Quedlinburg, Germany). *J. Chromatography*, 1960, **3** (5), 501–502 (in German).—A detailed description, with circuit diagram, is given of the apparatus, which is based on the principle of the oxy-hydrogen gas voltammeter.

S. M. MARSH

385. New type of tank for paper micro-electrophoresis of proteins from biological fluids. A. Seniów (Dept. of Parasitol. and Parasitic Diseases, High School of Agric., Wroclaw, Poland). *J. Chromatography*, 1960, **3** (6), 586–588.—The electrode vessels are slotted cylinders which can be rotated horizontally about their axes. The wetted paper strips can easily be stretched between the vessels before the samples are applied.

A. R. ROGERS

386. Apparatus for the direct photometry of lipid electropherograms. R. Ya. Vўsotskiĭ and V. F. Baumgart. *Voprosў Med. Khim.*, 1959, **5** (5), 377–380; *Ref. Zhur., Khim., Biol. Khim.*, 1960, (7), Abstr. No. 8867.—The apparatus is based on the use of reflected light for estimating the colour intensity of the electropherograms. The light absorption is measured with a photo-conductive element, which has a number of advantages over a conventional photo-cell. A check of the readings from the apparatus against the amount of lipids has shown that direct photometry of the electropherograms with a small range of colour intensities is possible. The results of determinations show good agreement with those of the elution method.

C. D. KOPKIN

387. Starch electrophoresis. III. Starch-gel electrophoresis. H. Bloemendal (Dept. of Biochem., Netherlands Cancer Inst., Amsterdam). *J. Chromatography*, 1960, **3** (6), 509–519.—A review is presented of methods and applications, with 63 references (*cf. Anal. Abstr.*, 1959, **6**, 5035; 1960, **7**, 4070).

A. R. ROGERS

388. Effects of field strength and ionic strength on velocity and spread of zones in starch-block electrophoresis. G. L. Miller (Quartermaster Research and Engng Center, Natick, Mass., U.S.A.). *J. Chromatography*, 1960, **3** (6), 570–575.—Fields that are too high and ionic strengths that are too low may cause disturbances in starch-block zone electrophoresis. With the apparatus used, best results were obtained with fields of <7·5 V per cm and at ionic strengths of >0·1. There is no significant direct relation between spread of zones and diffusion or distance of migration.

A. R. ROGERS

See also Abstracts—**123**, Un-impregnated cellulose paper for anions. **177**, Relative detector response in gas chromatography. **397**, Application of i.r. analysis to gas-chromatographic fractions.

Optical

389. The Ebert spectrograph. Anon. *Hilger J.*, 1960, **6** (2), 29–32.—Comparison of the Ebert and the Wadsworth spectrographs, in the light of recent modifications to the Ebert mounting, show the Ebert to be almost equivalent to the Wadsworth in performance, and superior in that it is mechanically simpler, has a greater wavelength range and can be adapted for direct reading. The instrument illustrated is a grating spectrograph of high definition and resolution, capable of analysing such materials as high-alloy steels, uranium and transuranic elements. The overlapping high-order spectra are separated by the "order sorter" described.

E. G. CUMMINS

390. Spectrographic electrodes for refractory samples. M. Slavin (Chem. Dept., Brookhaven Nat. Lab., Upton, Long Island, N.Y., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (3), 82.—The refractory components, particularly CaO and Al_2O_3, of a sample tend to be lost by mechanical ejection of the

residual bead by the arc stream. A standard drill bit forms a cup that is too shallow and it is claimed that a deep 30° conical crater, cut with a flat reamer, will hold the bead until it is completely volatilised into the arc discharge, thereby giving much improved reproducibility for these elements.

P. T. BEALE

391. Controlled-atmosphere excitation chamber for d.c. arc analysis. M. P. Brash and J. P. Phaneuf (Res. and Advanced Dev. Div., Avco Corp., Wilmington, Mass., U.S.A.). *Appl. Spectroscopy*, 1960, **14** (3), 80–81.—Cyanogen bands and heavy background are eliminated by arcing in an argon-oxygen atmosphere. The construction of a suitable quartz vessel for arcing in a nitrogen-free atmosphere is described in detail. P. T. BEALE

392. The use of scatter diagrams in emission spectroscopy. G. Holdt and A. Strasheim (Max-Planck-Inst. für Metallforschung, Stuttgart, Germany). *Appl. Spectroscopy*, 1960, **14** (3), 64–72.—The correlation and regression of two variables, such as the internal standard and analysis lines for the calculation of an intensity ratio, may be best shown by plotting one against the other. The statistical information is derived from the resulting scatter diagram and from a contour ellipse, constructed so that 10% of the points fall outside or on the ellipse perimeter. Many worked examples of the application of the method to different spectrographic problems are given. P. T. BEALE

393. The additive standard method in flame spectrophotometry, with special reference to the alkaline earths. G. E. Harrison (Radiobiological Res. Unit, Harwell, Berks., England). *Photoelect. Spectr. Gr. Bull.*, 1959, (12), 327–328.—A Unicam flame spectrophotometer is used to determine Ca, Sr, Ba and Mg in biological material. The interference effects of the large amounts of Na, K and P present are overcome by making standard additions of the elements being determined to the sample itself. An example is given of the determination of Mg in a bovine serum. P. T. BEALE

394. Photo-electric method for the measurement of spectra of light sources of rapidly varying intensities. H. H. Seliger (McCollum-Pratt Inst., Johns Hopkins Univ., Baltimore, Md.). *Anal. Biochem.*, 1960, **1** (1), 60–65.—The method depends on the use of photomultiplier tubes. Application is made to measurement of the luminescence of luminol (2-aminophthalhydrazide), and of H_2O_2 in the presence of Cl. H. F. W. KIRKPATRICK

395. Improved adaptation of Beckman model DU spectrophotometer for use with micro-litre absorption cells. D. Glick and L. J. Greenberg (Dept. of Physiol. Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *Anal. Chem.*, 1960, **32** (6), 736.—Improvements in certain details of an adaptation of the Beckman DU spectrophotometer for use with micro-litre absorption cells, reported previously (Glick and Grunbaum, *Ibid.*, 1957, **29**, 1243), are described. K. A. PROCTOR

396. Ultra-violet spectroscopy with polarised light. F. Dörr and M. Held (Phys.-chem. Inst. der Tech. Hochsch., München, W. Germany). *Angew. Chem.*, 1960, **72** (9), 287–294.—This review of the methods of steric orientation of particular electron oscillators in organic molecules stresses the additional structural information that is made available. The methods described are not confined to crystal studies. Work with otherwise orientated molecules, either confined in thin films, as with polymethine-cyanine on a poly(vinyl alcohol) foil, or as a streaming layer between glass plates (*e.g.*, pseudoisocyanine and tobacco mosaic virus), or aligned by molecular attraction in a monomolecular layer, as with hydrophobic and hydrophilic groups, is included. External electric and magnetic fields have also been used to produce orientation and thus facilitate the measurement of the alignment of electron oscillators with the aid of polarised light. The application of the method to smaller molecules is made simpler by statistical consideration of the number of similarly orientated molecules in a solid solution (*e.g.*, ice), a state in which they are orientated at random. Both photochemical and fluorescence studies have produced results with this approach. (88 references.) E. G. CUMMINS

397. Micro-quantitative infra-red analysis: application to gas-chromatographic fractions. J. E. Stewart, R. O. Brace, T. Johns and W. F. Ulrich (Beckman Instruments Inc., Fullerton, Calif., U.S.A.). *Nature*, 1960, **186**, 628–629.—Two i.r. measuring techniques for 20 to 100-μg amounts of material are described. (*i*) In the transmittance-ratio method for two-component mixtures, the transmittance is measured at two appropriate wavelengths and the log of the ratio is plotted against concn. of one component. (*ii*) In the extinction-ratio method, the ratio of the extinction at two wavelengths is plotted against the ratio of concn.; provided that there is no band overlap, the plot is rectilinear. Close control of cell thickness, solvent concn. or v.p. is unnecessary. The application of the methods to mixtures of *m*- and *p*-xylene, *m*- and *p*-cresol, 2,6-xylenol and *o*-cresol, and anthracene and phenanthrene are described in detail. G. S. ROBERTS

398. Potassium bromide capillary cell for infra-red micro-spectroscopy. E. D. Black (Pioneering Res. Div., Quartermaster Res. and Engng Center, Natick, Mass., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 735.—A method of preparing capillary cells by the potassium bromide pelleting technique, by embedding a wire in the pellet and then withdrawing it, is described. Such cells have good transmission from 2 to 15 μ. If necessary the sample may be recovered by dissolution of the pellet in water and solvent extraction. K. A. PROCTOR

399. Effects due to chemical state of the samples in X-ray emission and absorption. P. D. Zemany (Res. Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1960, **32** (6), 595–597.—Measurements made with a conventional X-ray spectrograph show that the chemical state of an element can affect the structure and position of an absorption edge and both the wavelength and the intensity of an emission line. These effects may be useful in determining the valency state of an element. K. A. PROCTOR

400. Electron-probe methods of X-ray micro-analysis. P. Duncumb (Cavendish Lab., Cambridge, England). *Brit. J. Appl. Phys.*, 1960, **11** (5), 169–176.—Applications are described of the electron probe as a point source of X-rays for analysis by absorption, fluorescence and emission spectroscopy. For the examination of surfaces for which emission spectroscopy is the most suitable method, the probe excites the specimen itself. With this method a resolving power of 1 μ and a detection sensitivity

of 10^{-14} g are attainable, and a scanning technique based on a selected emission line enables the distribution of a particular element to be presented visually.   G. SKIRROW

401. New automatic X-ray fluorescence spectrometer. J. R. Stansfield (Hilger & Watts, 98 St. Pancras Way, London, England). *Hilger J.*, 1960, **6** (2), 23–28.—Planned programming by patchboard, for the determination of up to 20 elements, is possible with the instrument described. The choice of a sequential system has made pulse-height analysis economical and therefore digital to analogue conversion is unnecessary. The choice of the many independently adjustable parameters provided, together with the selection of a patchboard to programme the angle of analysis to the nearest fiftieth of a degree, and other factors, permits automatically typed-out intensities to be presented in tabular form as they are measured. It is emphasised that the method is most suitable for the determination of major constituents and that, for example, different types of steel will need different calibration curves. Comparison with the direct-reading u.v. spectrograph is discussed. The X-ray calibration curve for nickel is a straight line through the origin with a standard deviation of 0·045% over the 8 to 16% range.   E. G. CUMMINS

402. Photo-electric automatic polarimeters. A. Emmerich. *Zucker*, 1960, **13**, 148–153.—The determination of sucrose with the photo-electric polarimeter and the standardisation of the polarimeter are described. Owing to rotation dispersion, the reading by yellow - green mercury light (5461 A) with the normal sugar soln. is 0·18% higher than with the normal control quartz plate. Preliminary results for the ratios between readings with yellow sodium light and with yellow - green mercury light for soln. of several raw sugars and molasses indicate an increase in the rotation dispersion of the molasses. Three automatic polarimeters are described.   SUGAR IND. ABSTR.

403. The ETL-NPL automatic polarimeter, type 143A. E. F. Dawson (Ericsson Telephones Ltd., Basford, Notts., England). *Photoelect. Spectr. Gr. Bull.*, 1959, (12), 315–320.—The split-field of the conventional instrument is replaced by a single field, but the half-shade principle is virtually retained by time-sharing by use of a Faraday cell, in which the twist in the plane of polarisation of light passing through the dense glass core is directly proportional to the current produced in the surrounding solenoid. A photomultiplier and feed-back principles in the electronic circuits have been used to produce an automatic instrument with a range of ±0·5° of arc and a minimum detectable rotation of one ten-thousandth of a degree.   P. T. BEALE

Electrical

404. A semi-automatic polarograph. G. W. Drake and C. B. Johnston (Univ. of Houston, Texas, U.S.A.). *J. Chem. Educ.*, 1960, **37** (5), 240–242.—With the instrument described, the effect of voltage change is plotted manually point by point on a chart from readings on a micro-ammeter. As the chart and voltage changer are coupled, voltage calibration is straightforward and operation simple. All the parts are readily available.
                                         E. G. CUMMINS

405. Polarographic determination of high concentrations of elements. L. N. Lyubimova and V. G. Sochevanov. *Zavod. Lab.*, 1960, **26** (6), 703–707.—Polarography of high concn. (0·005 to 0·25 M) can be carried out without loss of accuracy if the electrical resistance of the mercury in the capillary is reduced sufficiently. The capillary electrode described consists of a short length of capillary tubing (10 to 12 mm) fused to a wider tube carrying a platinum contact. Above the contact is fused a longer capillary tube to give the necessary mechanical resistance to the flow of mercury. Applications to the determination of Cu, Pb and Zn in ores are described.   G. S. SMITH

406. pH measurement at low temperatures using modified calomel and glass electrodes. L. van den Berg (Div. of Appl. Biol., Nat. Res. Council, Ottawa, Canada). *Anal. Chem.*, 1960, **32** (6), 628–631.—Modified reference and glass electrodes are described, suitable for pH measurements at temp. down to −30°. Polyhydric alcohols (or some of their derivatives) are added to the satd. KCl soln. of the calomel electrodes; the glass membranes of the glass electrodes are enlarged, and the inner buffer is replaced by mercury. Some preliminary results of measurements on frozen salt soln., milk and meat are given.   K. A. PROCTOR

407. Differential electrolytic potentiometry. III. An examination of the variables of the method applied to inactive reductants. E. Bishop (Washington Singer Lab., The University, Exeter, England). *Analyst*, 1960, **85**, 422–431.—A study is made of the electrical variables in the differential electrolytic potentiometry of irreversible reductants, *i.e.*, applications in which the electrolytic anode is inactive. For stated reasons the determination of hydrazine with $KBrO_3$ was selected as a test reaction. The effects of isolating individual electrolysis electrodes, varying the current with fixed electrode area, reducing the size of the electrodes to the micro and ultra-micro scale, varying the electrode area with fixed current and varying the source potential and stabilising series resistance of the current are examined. Experimental design criteria are formulated for the application of differential potentiometry to reactions of the type studied.
                                         A. O. JONES

408. New approach to radioassay of aqueous solutions in the liquid scintillation spectrometer. D. Steinberg (Nat. Heart Inst., Nat. Inst. of Health, Bethesda, Md.). *Anal. Biochem.*, 1960, **1** (1), 23–39.—The method described is based on a two-phase system, the solid phase consisting of finely divided phosphor and the liquid phase containing the radioactive material to be assayed. A number of solid phosphors, including a plastic scintillator, have been investigated and highly purified anthracene appears to be the most effective. The sample soln. is pipetted on to crystals of anthracene in a standard counting bottle and the fluorescent emission is counted in a liquid scintillation spectrometer. The method is applicable to virtually any compound soluble in water, salt soln., dil. acid or dil. alkali, and as no chemical conversion of the sample is required it can be recovered unaltered for other investigations.   R. A. BRENAN

See also Abstract—**263**, Carboxymethylcellulose gel for electrophoresis.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list.   When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	.	a.c.
ampere .	.	amp.
Ångström unit	.	A
anhydrous .	.	anhyd.
approximate, -ly	.	approx.
aqueous	.	aq.
atmospher-e, -ic .	.	atm.
boiling-point	.	b.p.
British thermal unit	.	B.Th.U.
calorie (large)	.	kg-cal.
calorie (small)	.	g-cal.
centimetre .	.	cm
coefficient .	.	coeff.
Colour Index	.	C.I.
concentrated	.	conc.
concentration	.	concn.
constant	.	const.
crystalline .	.	}cryst.
crystallised	.	
cubic	.	cu.
current density .	.	c.d.
cycles per second	.	c/s
density	.	ρ
density, relative .	.	d or wt. per ml
dilute	.	dil.
direct current	.	d.c.
distilled	.	dist.
ethylenediaminetetra-acetic acid	.	EDTA
electromotive force	.	e.m.f.
equivalent .	.	equiv.
gram	.	g
gram-molecule .	.	mole
half-wave potential	.	$E_{\frac{1}{2}}$
hour	.	hr.
hydrogen ion exponent	.	pH
infra-red	.	i.r.
insoluble	.	insol.
international unit	.	i.u.
kilogram	.	kg
kilovolt	.	kV
kilowatt	.	kW
liquid	.	liq.
maxim-um, -a	.	max.
melting-point	.	m.p.
microgram	.	μg (not γ)
microlitre .	.	μl
micromole .	.	μmole
micron	.	μ
milliampere	.	mA

milli-equivalent	.	milli-equiv.
milligram	.	mg
millilitre .	.	ml
millimetre	.	mm
millimicrogram .	.	mμg
millimolar	.	m$_M$
millivolt :	.	mV
minute (time) .	.	min.
molar (concentration)	.	M
molecul-e, -ar	.	mol.
normal (concentration)	.	N
optical rotation .	.	α_t^l
ounce	.	oz
parts per million	.	p.p.m.
per cent.	.	%
per cent. (vol. in vol.)	.	% (v/v)
per cent. (wt. in vol.)	.	% (w/v)
per cent. (wt. in wt.) .	.	% (w/w)
potential difference	.	p.d.
precipitate (as a noun)	.	ppt.
precipitated	.	pptd.
precipitating	.	pptg.
precipitation	.	pptn.
preparation	.	prep.
qualitative, -ly	.	qual.
quantitative, -ly	.	quant.
recrystallised	.	recryst.
refractive index	.	n_t^l
relative band speed	.	R_F
relative humidity	.	r.h.
revolutions per minute	.	r.p.m.
saponification value .	.	sap. val.
saturated calomel electrode	.	S.C.E.
second (time) .	.	sec.
soluble	.	sol.
solution .	.	soln.
specific gravity	.	sp. gr.
specific rotation	.	$[\alpha]_\lambda^t$
square centimetre	.	sq. cm
standard temp. and pressure	.	s.t.p.
temperature	.	temp.
ultra-violet	.	u.v.
vapour density .	.	v.d.
vapour pressure	.	v.p.
volt	.	V
volume	.	vol.
watt	.	W
wavelength	.	λ
weight	.	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	.	$>$	less than .	.	$<$
not greater than	.	$\not>$	not less than .	.	$\not<$
is proportional to	.	$\propto$	of the order of, approximately		$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, $e.g.$, Fe^{II}, Mo^V.   Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

ANALYTICAL ABSTRACTS

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